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Shear bond strength of zirconia orthodontic brackets depending on surface pretreatment of bonding base

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This study evaluated the shear bond strength (SBS) of zirconia orthodontic brackets under different surface pretreatment protocols to identify an optimal bonding strategy for clinical applications. Zirconia brackets (n = 94) were fabricated using 3 mol% yttria-stabilized zirconia powder and a ceramic injection molding technique. The bracket base was treated using one of the following: no treatment (Control), traditional alumina sandblasting (Group I), traditional alumina sandblasting with a 10-MDP-based primer (Group II), or silica-modified alumina sandblasting with a silane primer (Group III). Each group was divided into non-thermocycled and thermocycled subgroups. Brackets were bonded to the bovine enamel, and SBS was measured (10 brackets/subgroup). The failure mode was assessed using the Adhesive Remnant Index; and the debonded surfaces were analyzed by scanning electron microscopy and energy dispersive spectroscopy (2 specimens/condition). Within the limitations of this study, bracket base surface pretreatments significantly enhance the bonding strength of zirconia brackets. However, the combination of mechanical and chemical surface pretreatment results in excessive bond strength with a higher incidence of enamel damage upon debonding. Considering cost-effectiveness and procedural efficiency, the incorporation of traditional alumina sandblasting into the bracket base during manufacturing is recommended to ensure optimal bonding and safe debonding in clinical applications.

Keywords Orthodontic brackets, Shear bond strength, Zirconia brackets, Zirconia surface pretreatment

Ceramic brackets have been widely used in orthodontics since their introduction in the 1980s, offering an esthetic alternative to metal brackets[1]. However, conventional alumina-based ceramic brackets are prone to bracket wing fractures, which remains a significant drawback in clinical practice[2,3]. To address this issue, zirconia has emerged as a promising alternative due to its superior fracture toughness[4,5]. Furthermore, recent advancements in the composition, structure, and fabrication methods of zirconia have improved its esthetic appeal and mechanical performance, increasing its potential for orthodontic bracket applications[4–8].

Achieving a durable and reliable bond between brackets and dental hard tissue or prosthetic surfaces is a fundamental requirement for initiating orthodontic treatment with zirconia brackets. In restorative dentistry, various adhesion protocols have been developed to elevate the bond strength of zirconia to dental substrates. Accordingly, the incorporation of mechanical treatments and chemical adhesion promoters onto the zirconia surface is considered essential for good adhesion, in which either traditional alumina sandblasting combined with chemical promoters, such as 10-methacryloyloxydecyldihydrogenphosphate (MDP)-based products, or sandblasting with silica-coated particles associated with silane primers represents commonly used protocols with greater evidence in the literature[2,9,10]. While these techniques have been extensively studied for permanent zirconia bonding, their applications for orthodontic bracket bonding remain unclear, where bond strength must

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Test groups (n = 80)		Shear bond strength	Range
Non-thermocycled groups	Control	8.29 ± 1.64	6.25–11.03
	Group I	11.94 ± 1.98	8.90–15.04
	Group II	16.10 ± 3.73	11.06–22.63
	Group III	15.09 ± 3.43	10.56–19.67
Thermocycled groups	Control-T	4.92 ± 2.46	1.94–8.59
	Group I-T	9.51 ± 2.54	6.51–13.02
	Group II-T	12.98 ± 2.92	9.10–17.02
	Group III-T	10.21 ± 2.92	6.17–15.50

Table 1. Shear bond strength of all experimental groups. Mean ± Standard deviation (MPa).

Source	Type III sum of squares	df	Mean square	F	*p*
Surface pretreatment	691.938	3	230.646	29.820	$p < 0.001$
Thermocycling	237.937	1	237.937	30.762	$p < 0.001$
Surface pretreatment protocol x Thermocycling	16.144	3	5.381	0.696	0.558
Error	556.896	72	7.735		
Total	11,412.261	80			
Corrected total	1502.914	79			

Table 2. Two-way ANOVA for assessing the effect of bracket base surface pretreatment and thermocycling on the shear bond strength. *Statistically significant at $p < 0.05$. R square = 0.629; Adjusted R square = 0.593.

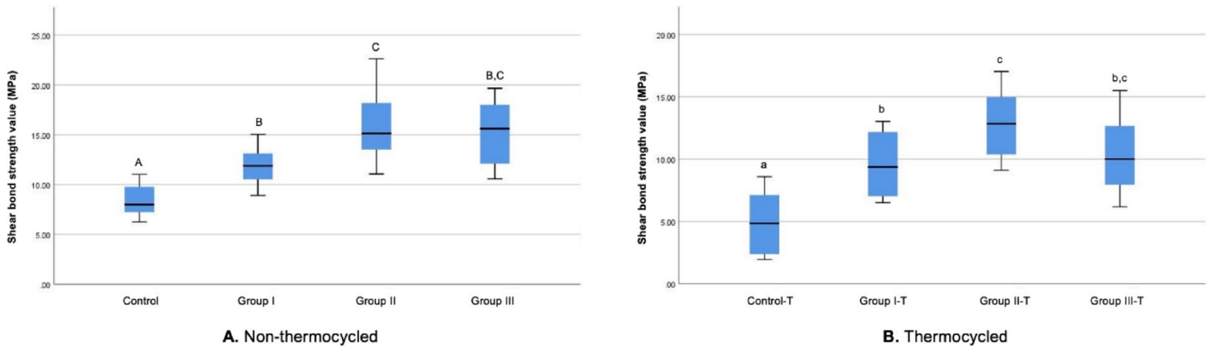

Fig. 1. Comparison of shear bond strengths among non-thermocycled groups (**A**); and thermocycled groups (**B**). The same superscript letters indicate no significant intergroup difference according to One-way ANOVA and post-hoc Tukey's honest significant difference test ($p < 0.05$).

be sufficient to endure orthodontic forces and mastication throughout approximately two years of treatment, yet allow for damage-free debonding upon completion[11].

With the growing application of zirconia in orthodontic bracket fabrication, the development of a reliable bonding protocol has become essential to ensure both adequate bond strength and safe bracket removal. Despite its clinical relevance, research on the adhesion characteristics of zirconia brackets remains limited. Accordingly, the aim of this study is twofold: firstly, to evaluate the effect of different common surface pretreatment protocols on shear bond strength (SBS) of zirconia orthodontic brackets, and secondly, to provide guidelines for manufacturing bonding base depending on surface pretreatment of the bracket base. The null hypothesis is that there was no significant difference in the SBS among surface pretreatment groups.

Results

Shear bond strength (SBS) tests and failure mode analysis

SBS values of all groups are presented in Table 1. The mean value of SBS was the lowest in Control-T. The effects of surface pretreatment, thermocycling, and their interaction on SBS were shown in Table 2. Both bracket base surface pretreatment and thermocycling had a significant effect on SBS ($p < 0.05$). However, no significant interaction effect between these factors was observed ($p > 0.05$).

Surface-pretreated groups observed significantly higher SBS values than Control, regardless of thermocycling ($p < 0.05$; Fig. 1). No significant difference in SBS value was observed between Groups I and III, and Groups II and III, as well as between Groups I-T and III-T, and Groups II-T and III-T ($p > 0.05$; Fig. 1). Additionally, SBS

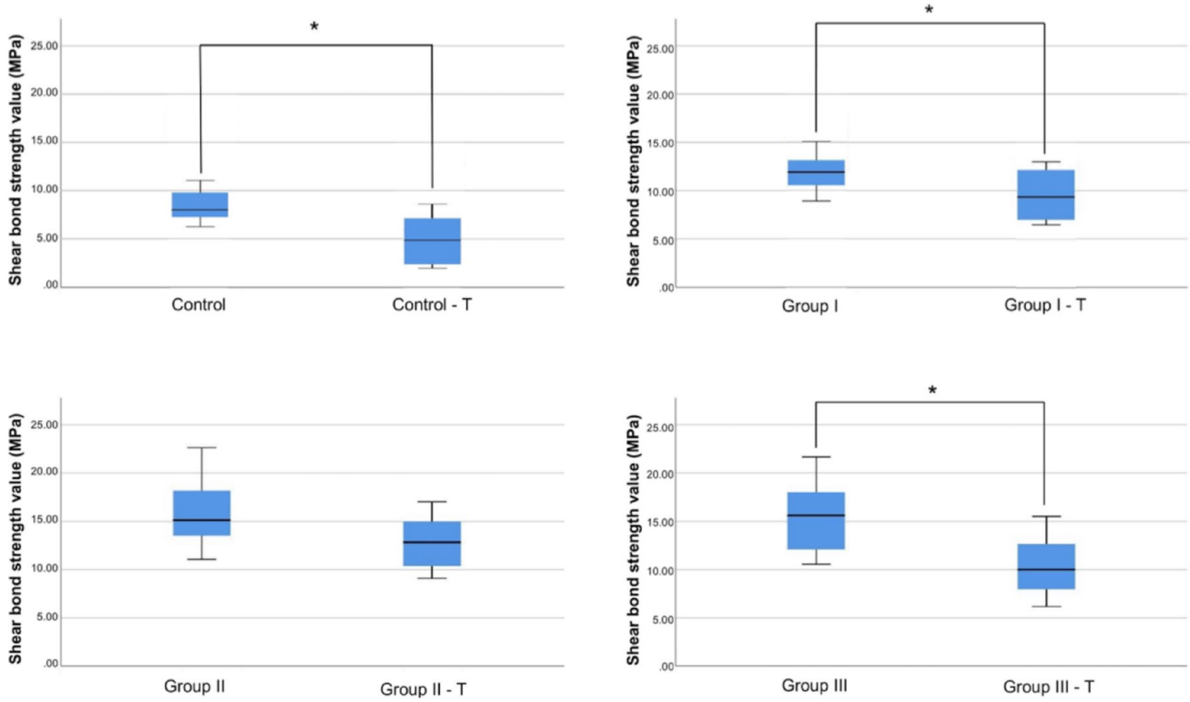

Fig. 2. Comparison of shear bond strengths between non-thermocycled and thermocycled groups. *The superscripts indicate differences in SBS according to independent t-test ($p < 0.05$).

ARI scores		0	1	2	3
Non-thermocycled groups	Control	–	–	2	8
		–	–	(20%)	(80%)
	Group I	–	1	2	7
		(0%)	(10%)	(20%)	(70%)
	Group II *	9	1	–	–
		(90%)	(10%)	(0%)	(0%)
	Group III	1	3	1	5
		(10%)	(30%)	(10%)	(50%)
Thermocycled groups	Control-T	–	–	2	8
		(0%)	(0%)	(20%)	(80%)
	Group I-T	–	1	2	7
		(0%)	(10%)	(20%)	(70%)
	Group II-T *	5	4	1	–
		(50%)	(40%)	(10%)	(0%)
	Group III-T	1	5	–	4
		(10%)	(50%)	(0%)	(40%)

Table 3. Frequency distribution of Adhesive Remnant Index (ARI) scores after debonding. Values are presented as number (%). *The superscript indicates differences between groups according to the Kruskal—Wallis test and the Mann–Whitney test in ARI scores ($p < 0.05$).

values of Groups I and III did not differ significantly from those of a commercially conventional metal bracket in the supplementary experiments under the same conditions. (Supplementary Table).

Although thermocycling decreased SBS significantly in Control, Group I and III ($p < 0.05$; Fig. 2), no significant change was noted in groups III ($p > 0.05$; Fig. 2).

Table 3 reports the Adhesive Remnant Index (ARI) for all groups, indicating the lowest ARI scores observed in Groups II and II-T ($p < 0.05$). Furthermore, ARI scores did not exhibit significant changes after thermocycling in any groups ($p > 0.05$).

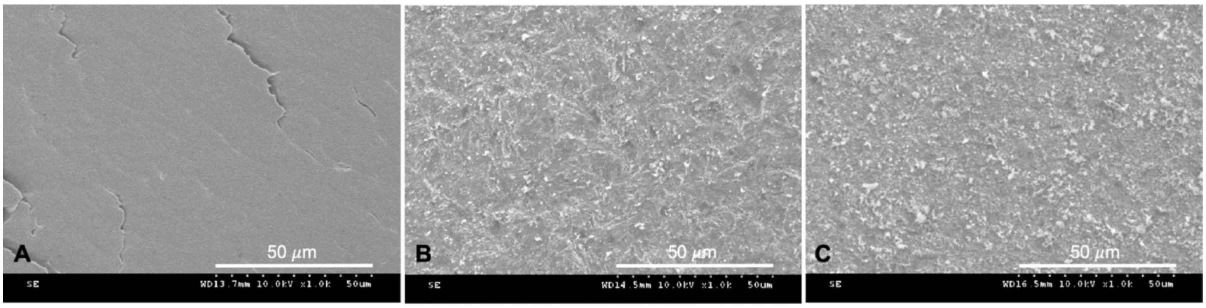

Fig. 3. Representative SEM images of bracket base at 10 kx: no treatment (**A**); after traditional alumina sandblasting (**B**); and after sandblasting with silica-modified Al_2O_3 particles (**C**).

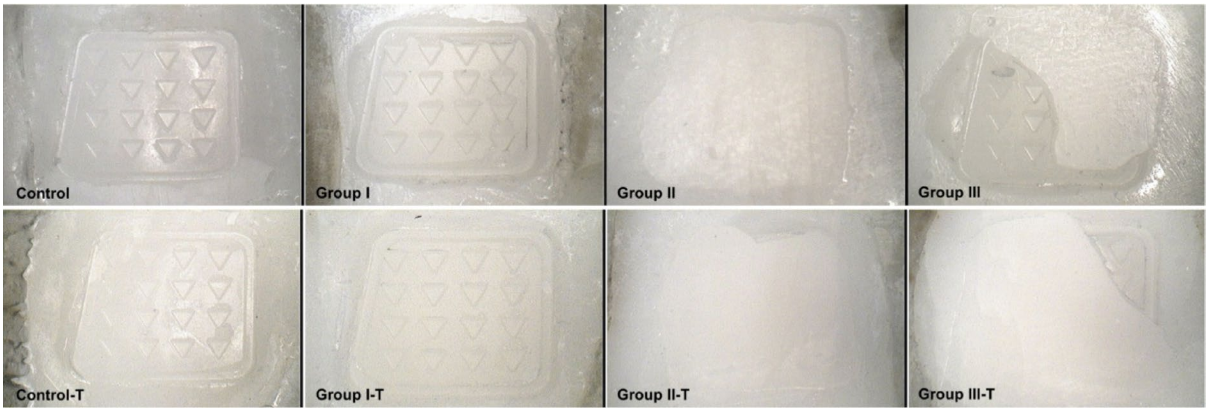

Fig. 4. Representative microscope images (50x) showing the failure mode on the enamel surface after debonding of all groups.

Scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) evaluation

Figure 3 illustrates the difference in the zirconia bracket base surface before and after sandblasting. A rougher surface microstructure was detected after sandblasting with either traditional alumina or silica-modified alumina particles.

Figures 4, 5, 6 illustrate the enamel and bracket base surfaces after debonding, revealing a typical adhesive failure at the bracket-adhesive interface presented in Control and Group I. In contrast, a higher incidence of failure at the enamel-adhesive interface was observed in Groups II and III. EDS analysis detected a high amount of hydroxyapatite over the bracket bases of Groups II and III, indicating enamel residue, while no calcium and phosphorus were identified on the bracket bases in the Control and Group I. Additionally, the debonded enamel surfaces of Groups II and III showed a mixture of grades 2 and 3 scores on the Enamel Damage Index, characterized by a rough surface with numerous coarse cratches and pronounced grooving.

Discussion

Previous studies have investigated the mechanical and optical properties of orthodontic zirconia brackets, comparing them to conventional polycrystalline alumina ceramic brackets. While 3 mol% yttria-stabilized zirconia (3Y-YSZ) bracket exhibited lower translucency than both polycrystalline alumina ceramic bracket products (e.g., 3 M™ Clarity™ Advanced) and highly translucent zirconia brackets containing 4–5 mol% yttria, it offers superior mechanical properties along with favorable optical stability. These induce greater tie-wing fracture resistance, smoother surface texture, and reduced frictional forces[6]. Given that the bonding characteristics of highly translucent zirconia materials is comparable to 3Y-YSZ[10,12,13], this study focused on evaluating various pretreatment protocols on the 3Y-YSZ bracket base surface. The goal was to provide evidence-based recommendations for manufacturing zirconia brackets with optimal bonding and debonding characteristics in clinical orthodontics. The finding reveals significant variations in the SBS values of zirconia brackets, depending on the bracket base surface pretreatment protocol. The null hypothesis was therefore rejected.

Several factors can influence the bonding ability of orthodontic brackets, such as the bracket material composition, bracket base design and base surface pretreatment methods, bonding surface characteristics and treatment methods, adhesive materials, and bonding protocols[11]. To ensure consistency, a single type of 3Y-YSZ bracket and one bonding protocol were used for testing. Additionally, ISO standards were applied the standardize the specimen preparation and shear bond strength testing, minimizing confounding variables and ensuring the reliability of the experimental results[14,15].

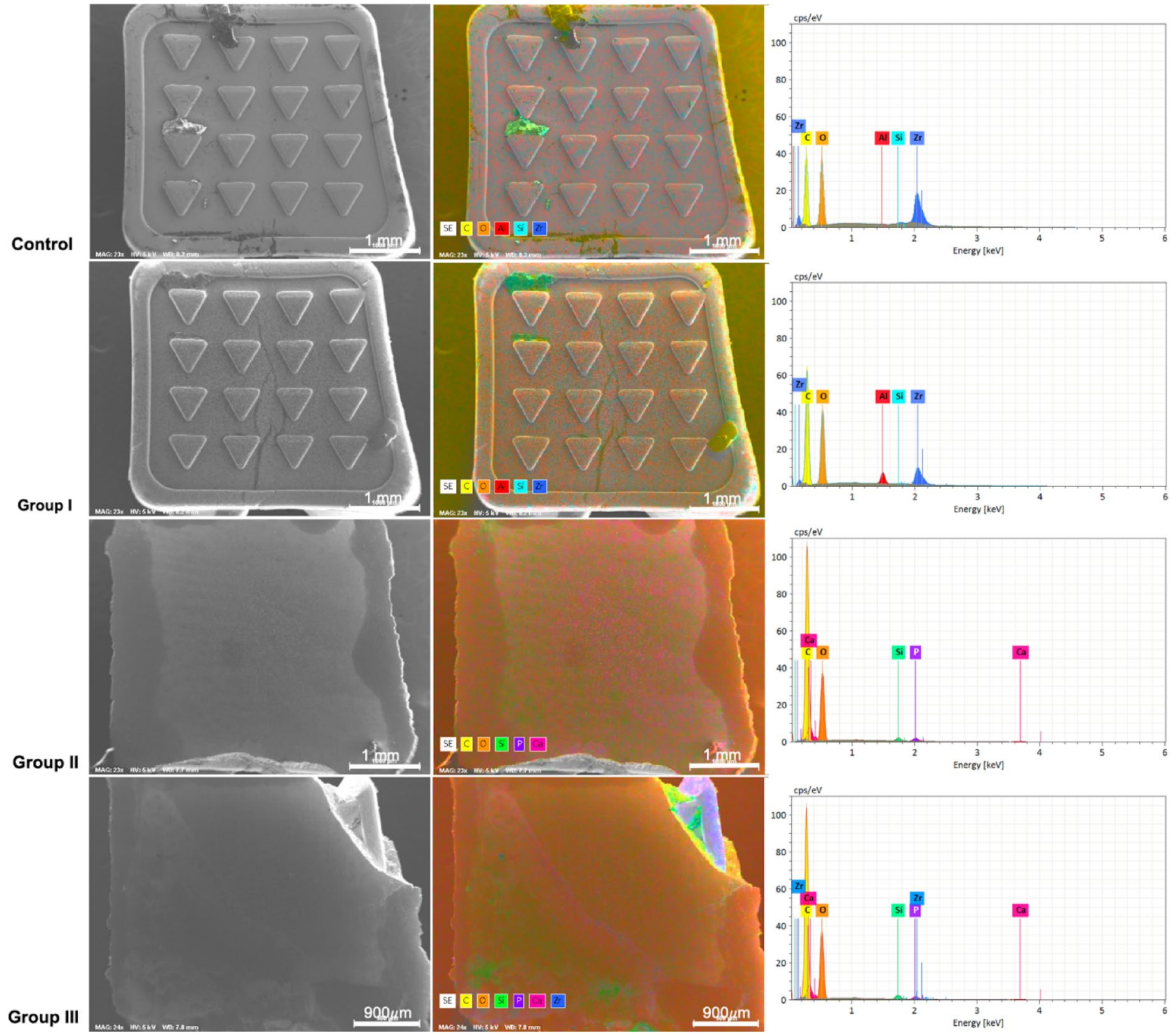

Fig. 5. Representative SEM and EDS images of the debonded bracket base with overlaid elemental maps showing the presence of calcium (Ca) on the bracket bases.

The primary requirement for bracket bonding is sufficient bond strength to resist orthodontic forces and masticatory load throughout active treatment without failure or detachment. Previous reports proposed that a bond strength range of 6–8 MPa is sufficient for successful clinical bracket bonding to enamel[16–18]. This range is broadly used as the standard reference value in the majority of in vitro bond strength investigations, even though it remains theoretical, and no consensus exists regarding the optimal bond strength necessary for reliable clinical performance of bonded brackets[19]. In this study, although the Control group with no surface pretreatment showed the lowest SBS values, it still reached an acceptable mean SBS of 8.29 MPa ($p < 0.05$; Table 1; and Fig. 1). However, thermocycling significantly reduced its SBS to 4.92 MPa, making its bond strength unreliable and insufficient (Fig. 2). In contrast, surface-pretreated groups demonstrated superior SBS values, with the thermocycled subgroups means ranging from 9.51 to 12.98 MPa (Table 1). Moreover, in the supplementary experiments, no significant differences were found in SBS values between Groups I and III and the commercially available metal brackets (Supplementary Table). Therefore, surface pretreatment of the bracket base is considered essential for a successful clinical bonding of zirconia brackets in this study.

Zirconia is a chemically inert material and lacks silica content, making etching impossible. To improve the adhesion between zirconia and resin cement, various mechanical and chemical surface treatments have been proposed[10]. This study employed three surface pretreatment protocols commonly cited in the literature. Sandblasting involves the propulsion of alumina particles at high speed, producing surface erosion and thereby generating a rough, clean, and more wettable surface. While this treatment sandblasting enhances zirconia surface roughness, the combined application of chemical promoters is essential to optimize allows an improvement of the adhesive performance[10,20,21]. For that purpose, 10-MDP-based primers are widely used. The 10-MDP molecule contains a phosphoric acid functional group that chemically interacts with zirconia, forming P-O-Zr bonds, while its vinyl terminal group enables copolymerization with the resin matrices. A hydrocarbon chain separates these reactive ends, imparting physicochemical properties such as viscosity,

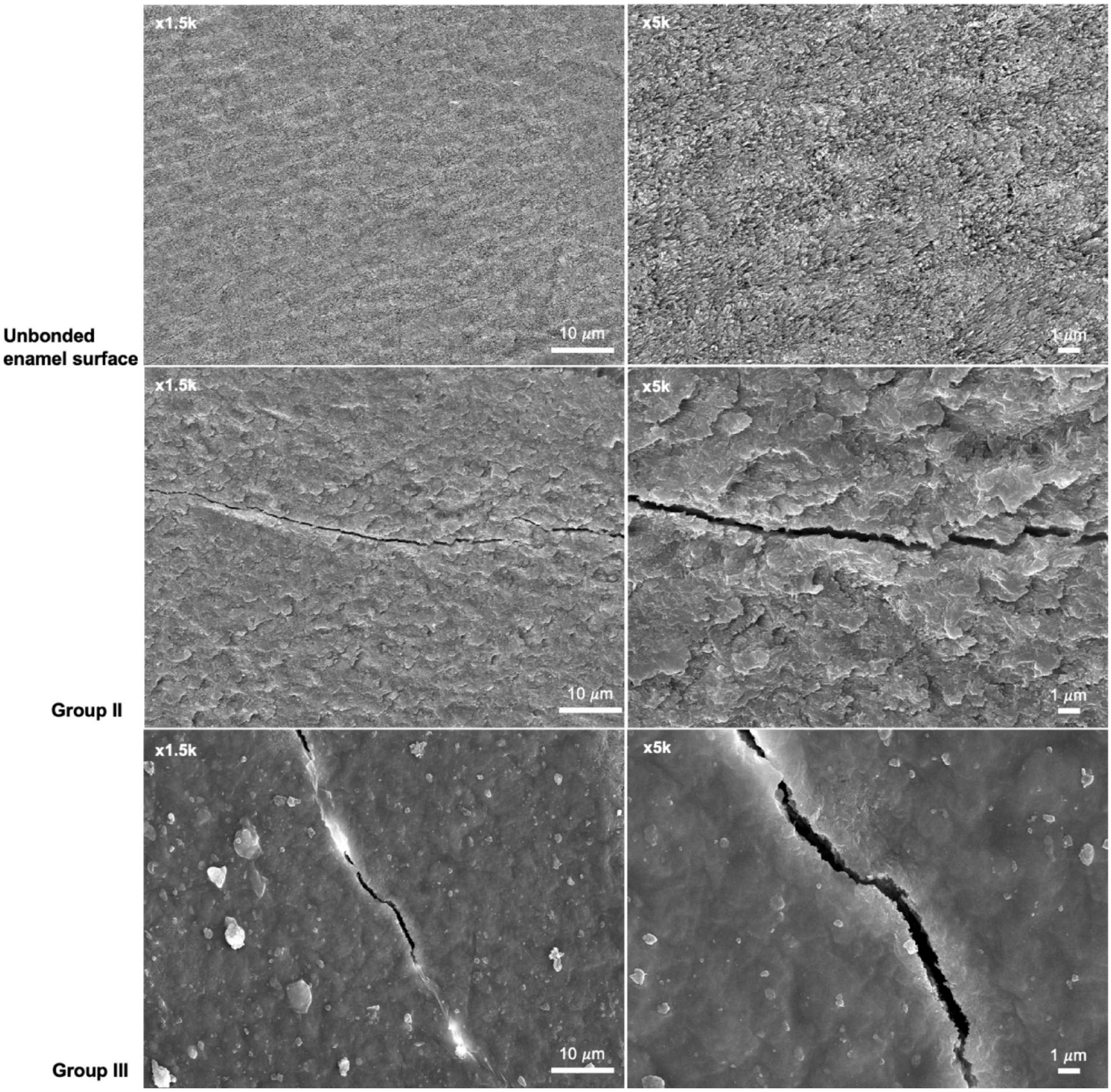

Fig. 6. FE-SEM images of unbonded and debonded enamel surfaces.

rigidity, hydrophobicity, and solubility. Consequently, 10-MDP-based primers have been shown to improve the bonding both with a self-adhesive composite (based on 10-MDP or other monomers) and with conventional composite cement[10]. Notably, although hydrolysis of the coordinate bond between MDP and zirconia has been implicated in the gradual degradation of the interface between MDP-conditioned yttria-stabilized tetragonal zirconia and methacrylate-based resins[22], MDP-containing primers have been reported to be able to ensure bonding stability after thermocycling of the zirconia surface treated with sandblasting[23–25]. As a result, Group II—treated with traditional sandblasting and MDP-containing primer—presented the highest and the most consistent SBS values, with no significant reduction after thermocycling (Table 1; Figs. 1 and 2). Another main approach to enhance zirconia adhesion is tribochemical silica-coating (TBS), in which alumina particles coated with silica are propelled against the surface[10]. This technique not only produces surface irregularities but also deposits silica, enabling the application of silane as a coupling agent to form bonds with both the resin composite and the silica layer. However, previous studies have shown that the silica deposition achieved with TBS may be incomplete, leading to an inconsistent silane-mediated coupling effect[26]. Such limitations could weaken the composite-zirconia interface and may explain the findings of the present study. Therefore, surface pretreatment of bracket base is considered essential for a successful clinical bonding of zirconia brackets.

The debonding behavior of the zirconia bracket is a crucial factor in ensuring the integrity of bonded enamel or prosthetic surfaces[11]. In this study, although group II provided the constantly highest SBS values among groups, it showed the lowest ARI score value with higher incidence of enamel damage ($p < 0.05$; Table 3; Figs. 4, 5, 6 and 7). Bond failures associated with low ARI scores typically occur near the enamel interface, posing a higher risk of compromising the enamel structure. Several studies have shown that higher bond strength in

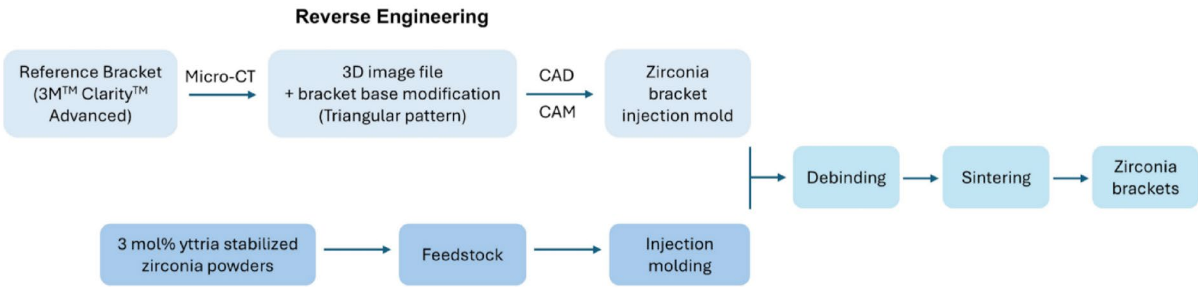

Fig. 7. Fabrication of zirconia orthodontic brackets using the ceramic injection molding technique.

ceramic brackets often results in bond failure at the enamel-adhesive interface as opposed to the bracket-adhesive interface[2,11,27]. To mitigate this problem, certain commercially available ceramic brackets have been engineered with built-in debonding features that facilitate bracket removal using specific pliers, thereby reducing enamel loss and minimizing chairside cleanup[28]. Nevertheless, these modifications introduce additional manufacturing complexity and cost, posing another consideration for both manufacturers and clinicians. Therefore, excessively high bond strength should be avoided, as it increases the incidence of enamel fracture or damage to existing restorations. For safe debonding, an optimal balance is necessary: adequate bond strength combined with adhesive failure and a high ARI score is more favorable[16]. In this study, significant difference in the SBS values was not observed between Groups I-T and III-T, and both presented mean SBS values above 9 MPa ($p > 0.05$; Fig. 1). However, EDS analysis indicated that Group I caused minimal damage to the enamel surface (Fig. 5). Taken altogether, group I appears to be the most favorable option, offering adequate bonding performance while preserving enamel integrity during debonding. Furthermore, this protocol requires solely mechanical surface pretreatment via traditional alumina sandblasting, eliminating the need for chemical application and thus offering advantages in terms of manufacturing efficiency and cost-effectiveness.

The challenge of achieving adequate orthodontic bracket bonding is considerably less than that in restorative dentistry[18]. Bonding to enamel is typically more durable and predictable than to dentin, primarily due to the enamel's lower water and organic content. Additionally, in orthodontics, the bond only needs to last for approximately two years of active treatment. Therefore, unlike permanent restorative procedures, the combination of a mechanical and chemical pretreatment to the base surface of the zirconia bracket may result in excessive bond strength, increasing the likelihood of enamel fracture upon debonding. Within the limitations of this study, incorporating traditional alumina sandblasting into the bracket base during the fabrication of zirconia brackets is recommended to enhance bonding effectiveness and facilitate safe debonding in clinical applications. Although the results of the present in vitro study provide valuable insight, they may not entirely reflect the complexities of the clinical environment, indicating the need for additional artificial aging protocols that more closely replicate intraoral conditions, as well as further in vivo investigations. In addition, detailed assessment of the enamel surface is necessary to better understand the extent of enamel damage that may occur during the debonding of zirconia brackets while considering the influence of different clinical debonding mechanisms. As the present study investigated the influence of bracket base surface pretreatment for bonding to enamel, additional investigations are needed to determine the bonding efficacy of zirconia brackets to various prosthetic materials.

Conclusions
Within the limitations of this study, bracket base surface pretreatments significantly enhance the bonding strength of zirconia brackets. The combination of mechanical and chemical surface pretreatment may result in excessive bond strength with a higher incidence of enamel damage upon debonding. Considering cost-effectiveness and procedural efficiency, the incorporation of traditional alumina sandblasting into the bracket base during manufacturing is recommended to ensure optimal bonding and safe debonding in clinical applications.

Methods
Specimen preparation
Zirconia brackets

Zirconia brackets for the maxillary right central incisors were manufactured using 3 mol% yttria-stabilized zirconia powder (Zpex; Tosoh Ceramic, Japan) through a ceramic injection molding technique (Fig. 7) with a polycrystalline alumina ceramic bracket (3 M™ Clarity™ Advanced, 0.022-inch slot) as described in previous study.[6] In the design process, the linear shrinkage of zirconia materials was accounted for to minimize discrepancies in final dimensions. Based on preliminary tests (data are not shown), the bracket base was designed with uniform triangle patterns to provide an initial mechanical interlocking to the adhesive (PTC Cero version 10.0.5.0, PTC Inc., USA), as shown in Fig. 8. This workflow demonstrates the potential to customize bracket designs—particularly the base—during the manufacturing stage, thereby enabling individualized bracket systems tailored to specific clinical setups, which is an additional benefit compared to using commercially available brackets.

A total of 94 zirconia brackets were prepared, of which 80 brackets were selected for the SBS tests, and 14 brackets were collected for scanning electron microscope (SEM) and energy dispersive x-ray spectroscopy (EDS) observation of the bracket base.

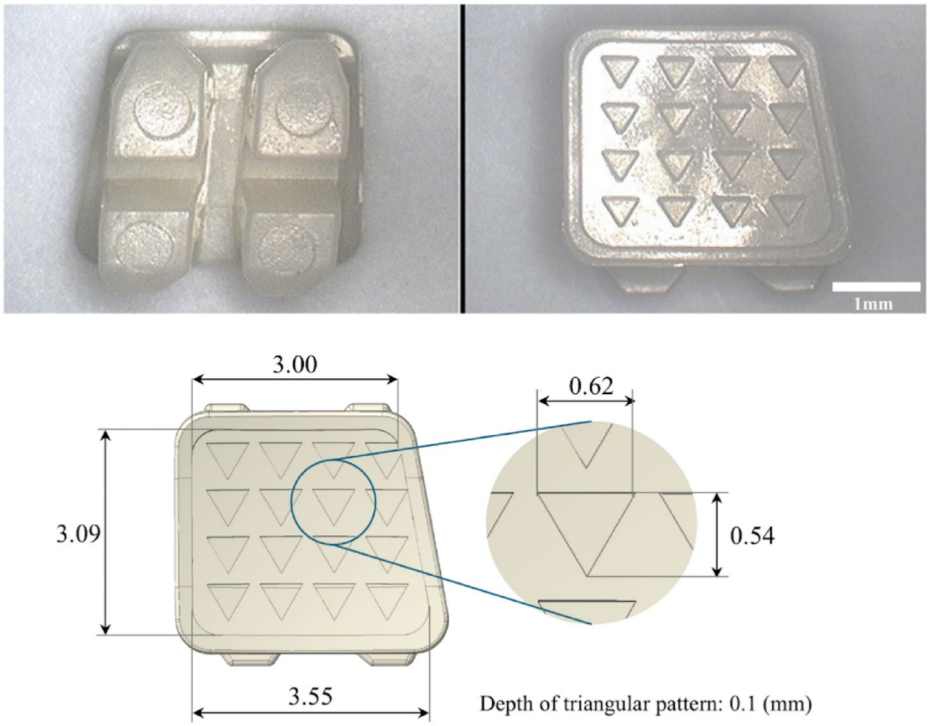

Fig. 8. Representative microscope images of the zirconia bracket body and base. (Unit: mm).

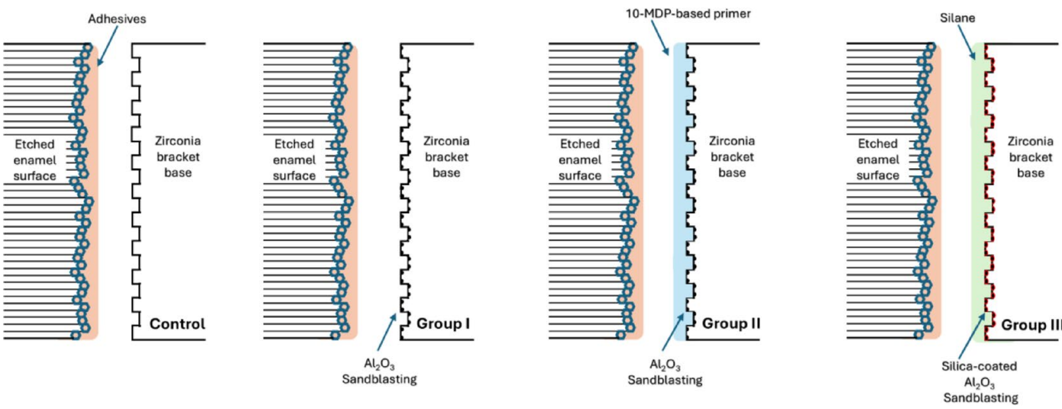

Fig. 9. Illustrations of etched enamel surface and zirconia bracket base depending on surface pretreatment protocols.

Before bonding, all bracket bases were thoroughly cleaned using 95% alcohol. The brackets were subsequently randomized into four experimental groups (n = 20 per group for SBS tests; and n = 2 per condition for SEM and EDS evaluation), each subjected to a different surface treatment protocol (Fig. 9).

- Control group: No treatment
- Group I (Air-abrasion): Bracket base surfaces were treated with conventional airborne-particle abrasion using 50-μ m aluminum oxide (Al_2O_3) particles for 10 s at a pressure of 40 psi. The particles were applied perpendicularly from a 10-mm distance.
- Group II (Air-abrasion + 10-MDP Primer): Brackets were sandblasted with 50-μ m Al_2O_3 particles. After sandblasting, the bracket bases were treated with a 10-MDP-based primer (Monobond Plus, Ivoclar Vivadent AG, Schaan, Liechtenstein).
- Group III (Air-abrasion + Silane Primer): Brackets were sandblasted with 30-μ m silica-modified Al_2O_3 particles (Cojet Sand, 3 M ESPE, Seefeld, Germany) and treated with a silane primer (RelyX Ceramic Primer, 3 M ESPE, St. Paul, MN, USA).

To assess the effect of aging on bond strength, each bracket group was further divided into two subgroups (n = 10 each), with one subgroup subjected to thermocycling (Table 4).

The sample size for SBS tests was determined based on a pilot study and power analysis. The minimum sample of 8 brackets in each subgroup was needed to detect a significant difference in SBS with 80% power, a significance level of 0.05, and an effect size of 0.65 using a one-way ANOVA test (G-power version 3.1.7; Franz Faul, Uni Kiel, Germany).

Enamel surfaces

Further specimen preparation was conducted according to the ISO standard[14]. In this study, bovine central incisor teeth of cattle with intact buccal enamel surface and root-resected were used as substitutes for human teeth in SBS testing[29–32]. The bovine teeth were obtained from cattle slaughtered for food processing purposes in accordance with Korean animal slaughter regulations at an authorized facility, with no relation to this study.

Prior to the bonding procedure, the prepared crowns were positioned in autopolymerizing acrylic resin (Polycoat EC-304, Aekyung Chemical, Chungnam, Korea) using a Teflon mold measuring 20 mm in diameter and 5 mm in thickness[31]. The specimens were immersed in cold water for five minutes to minimize heat generation during polymerization[29,33]. The enamel surface was then exposed by using a grinder-polisher machine (Ecomet 30, Buehler Ltd., Lake Bluff, IL, USA) and carbide papers (WetorDry Sheet, 800 and 1200-grit, 3 M, St. Paul, MN, USA) in wet condition[34]. After surface preparation, the specimens were kept in distilled water until bonding was performed.

Bonding protocol

The enamel surfaces were conditioned using 37% phosphoric acid gel (Reliance Orthodontic Products, Inc., USA) for 30 s, rinsed with water spray for 10 s, and subsequently dried using oil-free compressed air. Adhesive primer (Transbond™ XT, 3 M Unitek, Monrovia, USA) was then actively applied to the enamel surface using a rubbing motion[16].

Zirconia brackets were bonded to the enamel surfaces using a light-cured composite resin (Transbond™ XT, 3 M Unitek, Monrovia, USA). The adhesive was evenly applied across the bracket base, and any excess material around the bracket base was meticulously removed by an explorer. Polymerization was performed for 20 s using a light-curing unit operating within a wavelength range of 420–480 nm. Afterward, the specimens were immersed in distilled water until the commencement of the experimental procedures.

The testing protocol for this study is illustrated in Fig. 10. The stability of the bracket bonding was evaluated using thermocycling, an artificial aging process. According to ISO standard[15], a minimum of 5000 thermal cycles is recommended, and previous studies have shown that increasing the cycle count to 20,000 does not significantly impact the bond strength[35,36]. The application of 5000 thermal cycles is considered equivalent to simulating about six months of clinical function in the oral cavity[36]. Therefore, all thermocycled specimens were stored in distilled water at 37 °C for 24 h, followed by 5000 thermal cycles (5–55 °C) with a 15-s dwell time[10,35,37].

Measurements

Shear bond strength (SBS) tests and failure mode analysis

Ten brackets were examined in each surface prettreatment group and experimental condition (80 brackets in total, with 40 brackets for non-thermocycled groups and 40 brackets for thermocycled groups) (Table 4).

The debonding force was measured using a universal testing machine (Instron 3366; Instron Corp., USA) at a 1 mm/min crosshead speed. SBS (MPa) was calculated by dividing the load at failure (N) by the bonding area (mm^2).

Failure mode was assessed using a Microscope Image Analyzer (HIROX KH-1000, HIROX, Tokyo, Japan), and the amount of bonding resin remaining on the enamel surface was scored using the Adhesive Remnant Index (ARI) (Table 5).

Scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) evaluation of the bracket base after debonding

A random 14 brackets were selected for SEM and EDS evaluation of the bracket base.

Bracket bases before and after sandblasting being sandblasted with either 50-μ m Al_2O_3 particles or 30-μ m silica-modified Al_2O_3 particles were analyzed by using an SEM instrument (S-3000N, Hitachi, Tokyo, Japan) at a high magnification of 10 k to assess the surface alterations (n = 2 per condition; 6 brackets in total). Each specimen was affixed on SEM stubs and subjected to standard preparation, including freeze-drying with a freeze dryer (ES-2030, Hitachi, Tokyo, Japan) followed by platinum sputter coating to a thickness of 100 nm using an ion coater (E-1010, Hitachi, Tokyo, Japan). SEM imaging was conducted at an accelerating voltage of 15 kV.

SBS test groups (n = 80)		
Non-thermocycled (n = 40)	Thermocycled (n = 40)	Surface treatment of bonding base
Control (n = 10)	Control-T (n = 10)	No treatment
Group I (n = 10)	Group I-T (n = 10)	Traditional alumina sandblasting
Group II (n = 10)	Group II-T (n = 10)	Traditional alumina sandblasting + 10-MDP-based primer
Group III (n = 10)	Group III-T (n = 10)	Sandblasting with silica-modified Al_2O_3 particles + Silane primer

Table 4. Characteristics of SBS testing groups.

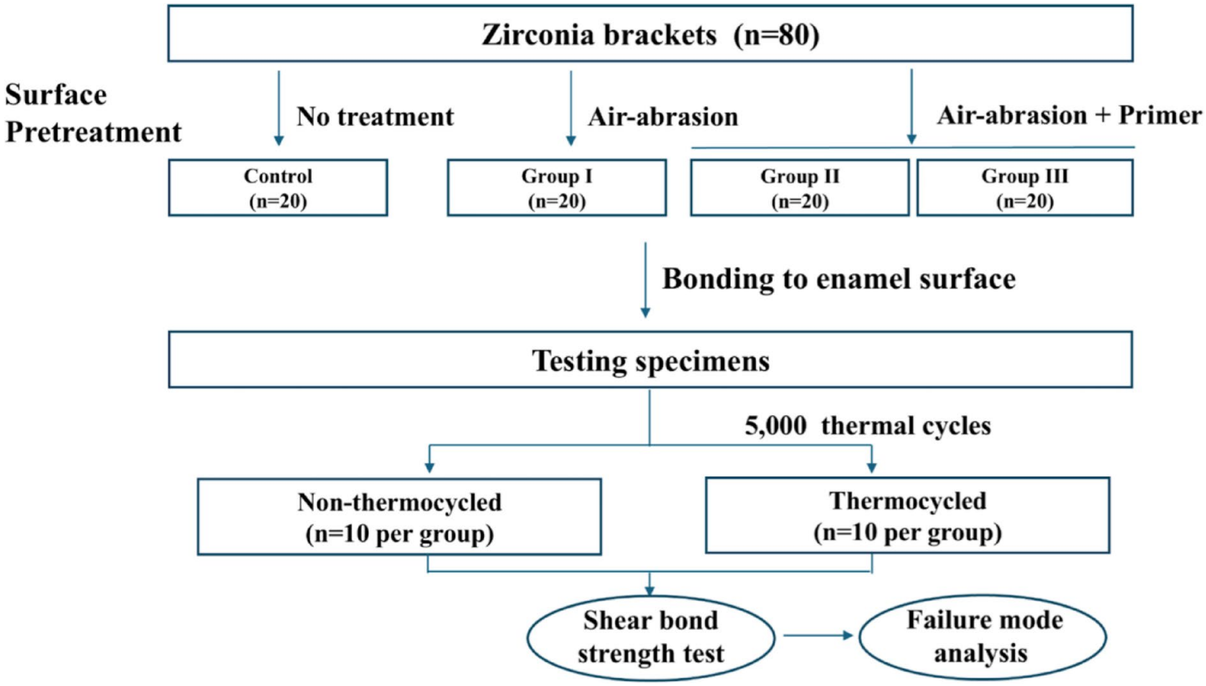

Fig. 10. A diagram showing the SBS testing procedure in this study.

ARI score	Criteria
0	No adhesive remained on the tooth surface
1	Less than half of the adhesive remained on the tooth surface
2	More than half of the adhesive remained on the tooth surface
3	All the adhesive remained on the tooth surface

Table 5. Adhesive Remnant Index (ARI) score.

To verify the risk of enamel damage after debonding, the a random 8 debonded bracket base surfaces (n = 2 per surface pretreatment group) were assessed by using SEM (MERLIN, Carl Zeiss, Oberkochen, Germany) in conjunction with Bruker EDS System. The presence of residual calcium (Ca)—indicative of enamel fragments—was investigated via elemental mapping[38]. Specimens (n = 2 per group) were fixed on conductive carbon tape, then coated with a thin carbon film using an ion sputter coater (EM ACE 600). EDS analysis was conducted using a XFlash®5060FlatQUAD (Bruker, Germany) at 15 kV accelerating voltage, 2000 × magnification, and a data acquisition time of 3000 s (X-flash 6, BRUKER, with ESPRIT 2.1 software).

Field emission scanning electron microscope (FE-SEM) analysis of the enamel surface
Representative debonded enamel samples from Groups II and III were selected following the debonding procedure (n = 2 per group), and compared with an unbonded buccal enamel surface prepared using the same above protocol. Specimens were sectioned, mounted on SEM stubs, dried, and sputter-coated with a 100-nm platinum layer. The enamel surfaces were examined under a FE-SEM (MERLIN, Carl Zeiss, Oberkochen, Germany), and photomicrographs were acquired at an operating voltage of 15 kV with high magnifications.

Surface characteristics were assessed using the Enamel Damage Index, where: Grade 0 denotes a smooth surface without scratches and visible perikymata; Grade 1 indicates an acceptable surfcae with fine scattered scratches; Grade 2 corresponds to a rough surface with numerous coarse scratches or shallow grooves; and Grade 3 represents a severely damaged surface with pronounced groooves and enamel defects visiable to the naked eye[19,39,40].

Statistical analysis
The Shapiro–Wilk test was performed to evaluate whether the data followed a normal distribution. Two-way ANOVA and Tukey's honest significant difference post-hoc test were performed to evaluate the effects of bracket base surface pretreatment, thermocycling, and their interaction on the SBS. One-way ANOVA and post-hoc Tukey's honest significant difference test were performed to evaluate the significant difference in the SBS. Within each surface pretreatment group, an independent t-test was administered to determine changes in SBS. The Kruskal–Wallis test and the Mann–Whitney test were used to assess the statistically significant differences in

ARI scores between surface pretreatment groups. All statistical analysis was conducted with IBM SPSS 24.0 Statistic Software (IBM Co., Armonk, NY, USA). The statistical significance level was determined to be 0.05.

Data availability
The datasets used and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Received: 15 July 2025; Accepted: 26 September 2025
Published online: 03 November 2025

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Acknowledgements
This research was supported by a research grant from the National IT Industry Promotion Agency (NIPA) in Korea (S0901-25-1003). We thank Dentium Co., Ltd., Korea for providing the zirconia bracket specimens for this study.

Author contributions
Hai-Van Giap and Hyojin Kim have equally contributed as first authors. Hai-Van Giap, Hyojin Kim, and Kee-Joon Lee conceived and designed the project. Hai-Van Giap and Hyojin Kim performed research, conducted data analyses, and constructed the manuscript in consultation with Kee-Joon Lee. Kyung-Ho Kim, Hyung-Seog Yu, Jae-Sung Kwon, and Hyeonjong Lee revised the manuscript. All the authors read and approved the manuscript.

Declarations

Competing interests
The authors declare no competing interests.

Additional information
Supplementary Information The online version contains supplementary material available at https://doi.org/10.1038/s41598-025-22230-9.

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