Effects of Surface Treatments on Shear Bond Strength of Metal Bracket Bonded to Ultra-Translucent Zirconia

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Abstract

This study compared the shear bond strengths (SBS) of metal brackets bonded to treated ultratranslucent zirconia (UTZ) surface.

UTZ specimens were treated with 4 procedures: (1) No treatment (N), (2) sandblasting with 50 μ m Al₂O₃ (SB), (3) roughening with zirconia stone burs (DU), (4) roughening with fine diamond zirconia burs (DIA). The specimens were examined by non-contact profilometry and scanning electron microscopy to measure surface roughness. The metal brackets were bonded to UTZ specimens, and the half of each method was initially tested SBS and specified failure mode. Another half was aged with 5,000 thermal cycles before testing. The surface roughness data were analyzed with one-way ANOVA (p<0.05). The SBS data were analyzed using one-way ANOVA and the Tukey post hoc test (p<0.05) while the scores of failure mode were analyzed using Fisher's exact test.

UTZ treated with SB showed the highest surface roughness. The initial SBS of all was no significant difference. While the SBS of N was significantly decreased, and most of resin remained on the base of bracket when UTZ treated with DU after thermocycling. With limitation, roughening UTZ with zirconia stone burs not only had acceptable SBS but also was easier to remove resin.

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Introduction

The use of zirconia has become common esthetic due to increasing demands. biocompatibility, and the popularity of metal-free restorations¹. Bonding orthodontic appliances to zirconia crowns is challenging due to the inactive surfaces and the difficulty in creating microinterlocking at the surface². Chemical pretreatment with 10-MDP (10-methacryloxydecyl dihydrogen phosphate) containing primer or 10-MDP containing resin cement and airborne particles abrasion to increase surface roughness and wettability for bonding improvement is the most common protocol³⁻⁴.

Traditionally, dental zirconia was made of

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Dr. Preeya Suwanwitid, D.D.S., Higher. Grad. Dip. of Clin. Sc. (Orthodontics), Chulalongkorn University, Diplomate, Thai Board of Orthodontics, The Dental Council, Department of Orthodontics, Faculty of Dentistry, Chulalongkorn University, Henri-Dunant Road, Wangmai, Patumwan, Bangkok, Thailand. E-mail: Preeya.s@chula.ac.th fine tetragonal zirconia crystal with small amounts of yttrium stabilizers (3Y-TZP, 3 mol% yttria-stabilized tetragonal zirconia polycrystals). The current method to improve the translucency of 3Y-TZP is by increasing the yttrium content. Introducing 5% mol (5Y-PSZ), a partially stabilized zirconia, has been found to enhance translucency by introducing cubic (c) phase turn into tetragonal (t) phase⁵. The transition from the cubic (c) phase to other phases decreases the stress-induced transformation toughening that affected strength and toughness⁶. Thus, ultratranslucent zirconia (UTZ) has increasingly used especially in anterior teeth restorations.

Previous studies examining shear bond strength to zirconia surfaces, ceramic brackets and 3Y-TZP monolithic zirconia were commonly used⁷. Most of studies suggested modifying surface with sandblasting combined with 10-MDP primer could increase the shear bond strength⁸⁻⁹. Although intraoral sandblasting technique can perform directly to the zirconia crown surface, but this is sensitive technique and needs the protective equipment to protect patient and environment from the airborne particles¹⁰.

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Ultra-translucent zirconia is the new generation of monolithic zirconia that mechanical properties different from 3Y-TZP. are Furthermore, there have been few reports on the mechanical surface treatment of zirconia surfaces for bonding orthodontic brackets and the long term shear bond strengths (SBS). Thus, the objective of this study was to present and compare the initial and long term SBS of metal brackets bonded to UTZ after surface treatments, specifically sandblasting and roughening with zirconia burs.



Figure 1. The representative image of the captured area, where non-contact profilometry was used to evaluate roughness parameters, was reported as Ra (Roughness average).

Materials and methods

Preparation of UTZ specimens

Eighty UTZ specimens were divided into 4 groups based on the surface treatments, as shown in Table 1. The materials used in this study were listed in Table 2. The preparation of UTZ blocks followed the manufacturer's instructions. In this study, industrially manufactured LAVA Esthetics, a 3M ceramic disc (16 mm tall), was used. The pre-sintered UTZ disc was cut into cylindrical shapes using a milling machine (Bego, USA) at 1200 rpm and a cutting machine at 500 rpm (Buehler, USA., Lowspeed saw). This process was done to prepare

specimens with a height of 4 mm and 6 mm in diameter.



Figure 2. The SEM (500x magnification) images of UTZ surface of each group after surface treatment.



Figure 3. Results of SBS of all groups. Error bars indicated the standard deviation of each group. 0 = Initial SBS and 1 = long term SBS, * showed statistically significant between N0 and N1 groups. ** showed statistically significant between long term SBS groups.

The pre-sintered cylindrical shapes of UTZ were first sintered (1500 °C) for 65 minutes. Then, all specimens were glazed using GLAZE LT powder (VITA & Co. KG, Germany) and fired again at 850 °C for 90 minutes. To ensure consistent surface preparation, all steps were carried out by a single dental technician, who

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followed the manufacturer's instructions meticulously. After that cylindrical UTZ specimens were in PVC pipes and immersed in distilled water with ultrasonic vibration (5210, BRANSONIC, Germany) for 10 minutes and then dried.





Surface treatment procedures

In N group, 20 UTZ specimens were only in an ultrasonic cleanser with distilled water for 10 minutes. In SB group, the bonding area of 20 specimens was abraded using 50 μ m Al₂O₃ particles at 40 psi for 10 seconds. The sandblasting machine's tip end was positioned perpendicular to the bonding area, maintaining a distance of over 5 mm from the area.

In DU and DIA groups, the force calibration used during roughening was set at 5 N.⁵ In DU group, the bonding area was roughened using Dura-Green® DIA zirconia stone burs with constant water cooling. A micromotor (NSK ultimate 500, Japan) operated 10 seconds at 20000 rpm. In DIA group, the

bonding area was roughened using fine diamond burs with constant water cooling. An airotor (NSK Pana Max PAX TU B2, Japan) operating at 200000 rpm was used for 10 seconds. The roughening direction was set as a single forward direction from the right end to the left end. These procedures were done by the same operator who was calibrated to apply a force of 5 N.⁵ A new bur was used for each specimen. The heat generated during each polishing step was measured using a thermocouple. The thermal energy produced during each polishing stage was assessed using a thermocouple^{4,6,12}.



Figure 5. SEM images (40x magnification) of the debonded UTZ surface and the metal bracket surface of all groups after SBS testing. Abbreviations: Zr (UTZ), R (Resin cement), Br (Metal bracket).

Surface roughness measurement

After the surface treatments, UTZ specimens were cleaned with ultrasonic machine in distilled water 10 minutes and then dried. The surface roughness of the specimen was analyzed by a non-contact surface roughness tester (Infinitefocus, Alicona, Tokyo, Japan). The polarizing light of tester captured the surface and quantified its roughness using a dedicated program. The roughness parameter evaluated was the average surface roughness (R_a), calculated as the average of five readings.

Metal bracket placement

Metal brackets for maxillary right central incisors with a slot size of 0.018" x 0.022" (Tomy®, Japan) were placed at the straight line through the center surface of UTZ specimen. The

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adhesive bonding solution (Universal adhesive®, USA) was applied, followed by gentle air blowing for 20 seconds. A standardized force of 300 grams, applied with a Dontrix gauge (Orthopli, Philadipi, PA), was used during coated the bonding area (4 x 3 mm²) of the metal brackets with adhesive resin (Transbond XT, USA). Any excess adhesive resin was removed using a fine explorer and cured with an LED curing device (Mini LED III, Acteon®, France) for 10 seconds at each margin, maintaining a 45-degree angle to the surface. A radiometer (Kerr Corp., Orange, CA) was used to ensure that the light had a constant output of 1800 - 2200 mW/cm².

N0	No surface treatment		No surface treatment	
SB0	50 µm Al₂O₃ 40 psi sandblasting		50 µm Al₂O₃ 40 psi sandblasting	
DU0	Roughening with Dura-Green [®] DIA zirconia stone burs	DU1	Roughening with Dura-Green [®] DIA zirconia stone burs	
DIA0	Roughening with fine diamond zirconia burs	DIA1	Roughening with fine diamond zirconia burs	

Table 1. Groups of the different surface treatedUTZ in this study.

*0: specimens were immediately tested SBS, 1: specimens were aged with 5,000 thermal cycles before testing.

Materials/Trade name	Lot No.	Main component	Manufacturer
UTZ, 5Y-PSZ ceramic/LAVA Esthetic	6371590	5% Y ₂ O ₂ , 95% ZrO ₂ , alumina	3M ESPE, USA
Glaze materials	4639387	Vita AKZEN Plus	Vita, Germany
Universal adhesive®	80411A	Organophosphate monomer (10-MDP), dimethacrylate resins (BisGMA, etc.), HEMA, Vitrebond copolymer, filler, ethanol, waler, initiators, silane	3M ESPE, St. Paul, MN, USA
Transbond XT adhesive paste	St380977	Silane treated quartz, bis GMA, bisphenol A bis (2-hydroxyethyl ether), dimethacrylate, silane-treated silica	3M Unitek, Monorvial, CA, USA
Metal bracket (0.018", Omni arch Tomy®)	-	Nickel, Chromium	Japan

Table 2. Materials were used in this study.

ARI Score	Criterion
0	No adhesive remaining on UTZ
1	Less than half of the adhesive remaining on UTZ
2	More than half of the adhesive remaining on UTZ
3	All adhesive remaining on UTZ

 Table 3. Adhesive remnant index (ARI) score.

Shear bond strengths measurement

All UTZ specimens bonded metal brackets were incubated in a 37 °C for 24 hours. The SBS of 40 specimens (N0, SB0, DU0, DIA0)

was tested using a universal testing machine (EZ-S, SHIMADZU, Japan). The load was applied parallel to the zirconia-bracket interface in a gingival-occlusal direction, using a knifeedged rod at a rate of 1 mm/min until failure occurred, with a force of 50 N. The force required to debonded the bracket was recorded in Newtons, and the values were calculated as MPa. The other half of the speciemens (N1, SB1, DU1, DIA1) were tested SBS after undergoing thermocycling, which involved 5000 cycles between 5 and 55 °C. Each cycle consisted of a 30-second dwell time in water and a 5-second transfer time.¹¹

Surface treatment method	N (sample)	Mean (Rª)	SD
No surface treatment (N0/N1)	20	0.021	0.01
50 μ m A $_2O_3$ 40 psi sandblasting (SB0/SB1)	20	2.67	0.49
Dura-Green®DIA stone burs (DU0/DU1)	20	1.21	0.09
Fine diamond zirconia burs (DIA0/DIA1)	20	1.43	0.32

Table 4. R_a Surface roughness (μ m) after surface treatment.

*R_a is roughness average of five readings.

Failure mode determination

Failure mode was specified usina Adhesive Remnant Index (ARI) score developed by Årtun and Bergland (1984).¹² The failure modes between UTZ and metal bracket were ascertained under a 20x light microscope (SZ61, Olympus, Japan). The ARI score showed in Table 3 was a 4-point score used to evaluate the amount of adhesive remaining on the bracket base after debonding. The failure modes were categorized as adhesive failure at resin-bracket interface, mixed (adhesive + cohesive) failure and adhesive failure at zirconia-resin interface.

Surface Morphology

The surface morphology UTZ of specimens after surface treatments and SBS testing (one sample per group) was tested using a field emission scanning electron microscope (FE-SEM; SU-70, Hitachi, Tokyo, Japan) at a 500x. Additionally. magnification of the representative images of the debonded brackets from each group were observed using the same method.

Statistical analysis

All data were analyzed using SPSS at a significance level of α = 0.05. The roughness data were analyzed using one-way analysis of

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variance (ANOVA). The SBS data, which satisfied both the assumptions of normality and variance homogeneity, were analyzed using oneway ANOVA followed by the Tukey post hoc test. The ARI scores were analyzed using Fisher's exact test.

Ethical considerations

This research was laboratory procedure that all specimens did not involve human tissue. While we recognized the importance of upholding ethical standards in scientific research. Our research aligned with Dental Material Research and Development Center protocols of Dentistry Faculty, Chulalongkorn University. We believed in maintaining transparency and accountability in our research practices.

Results

The mean surface roughness values and the surface morphology were presented in the Table 4 and Figure 1. The surface roughness of treated surface values groups were significantly increased (p<0.05). The surface roughness of SB group was significantly the highest (p<0.05) and there was no significant difference between DU and DIA groups (p>0.05). The grooves and bridges of treated surface groups appeared to be irregularly distributed. In particular, the SEM image of SB group showed the microabraided surface, while DU and DIA groups showed the clearly groove on UTZ surfaces (Figure 2).

The mean SBS of all groups was represented in Figure 3. Initially, the SBS of all groups was insignificantly different (p>0.05). After thermocycling, only no surface treatment group (N1) was significantly decreased of SBS (p<0.05) but the SBS of treated surface groups were insignificantly different (p>0.05).

The frequency of different failure modes for all groups was represented in Figure 4. In no surface treatment groups (N0 and N1), the adhesive resin remained entirely on the bracket, and SEM images of the fractured area displayed 100% adhesive failure at the zirconia-resin interface (Figure 5).

In treated surface groups (SB0 and DIA0), 70% of the failure was adhesive at the resinbracket interface, while 30% was mixed failure. In DU0 group, the adhesive failure at the resinbracket interface was increased to 100%, and SEM images showed all resin outstandinging on

the zirconia surface, with a distinct impression of the bracket mesh (Figure 5).

After thermocycling, SB1 group showed 100% adhesive failure between resin-bracket surface and DU1 group showed 70% adhesive failure between zirconia-resin surface and 30% mixed failure. In DIA1 group showed 40% mixed failure and no adhesive failure between resinbracket surface.

Discussion

Orthodontists may encounter the patients who have UTZ crown that achieving strong bond must require chemical mechanical and bonding.14-17 Previous studies presented that SBS of orthodontic brackets were acceptable after various surface treatments with alumina sandblasting, tribochemical silica coating, coarse soft-lex disc roughening, and CO₂ laser.^{6,18-22,23} Although aluminum sandblasting is commonly recommended to improve the bond strength but intraoral sandblasting produced an aerosol contaminated with aluminum oxide particles and some of aluminum-allergic-patients may be concerned.^{10,24-25} Moreover, there has been few studies of long term SBS that roughening UTZ surfaces with dental burs.

Abrasion zirconia surface with more than 50 µm of particle size did not increase in bond strength, furthermore phase transformation and minor cracks could be induced which resulting a decreased of mechanical properties.²⁶⁻²⁸ However previous studies reported that surface roughness from sandblasting and roughening with zirconia stone burs had no effect on mechanical properties of zirconia crown.^{4,29}

The mean SBS of this study presented the values of all groups prior that to thermocycling were more than minimum bond strength of 5.9-7.9 MPa, which was acceptable for clinical use.³⁰⁻³¹ Although SBS was higher than 7.9 MPa, but this indicated initial SBS. Whilst duration of orthodontic treatment required at least 12-24 months, thus long term SBS should be considered.³² The half of specimens in this study was simulated bonding in the oral environment with 5,000 thermal cycles, which could be equivalent to 6 months in vivo.¹¹ Although SBS of no surface treatment UTZ was significantly decreased and less than minimum required, but SBS of surface treatments UTZ was still higher than minimum required. Thus, surface

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treatment UTZ was recommended for orthodontic bracket bonding.

Considering failure mode, the adhesive failure at the zirconia-resin interface was easier and safer for debonding. However, this failure mode was presented in no surface treatment groups but the long term SBS was clinically unacceptable. While sandblasting technique enhanced initial and long term SBS but adhesive failure mostly presented at the resin-bracket interface that clinicians must be careful when debonding or removing residual adhesive resin. For the roughening surface with dental burs, the adhesive failure at the zirconia-resin interface was mostly presented, especially in roughening with zirconia stone bur that adhesive resin was mostly remained on bracket base after thermocycling. Thus, roughening with dental burs especially zirconia stone bur was easier to remove adhesive resin on UTZ surface.

The limitations of this study were that SBS was obtained by laboratory procedures, although these results presented acceptable SBS for clinical use, but clinicians should carefully apply in clinic. Furthermore, this study simulated long term intraoral condition which may be equivalent to 6 months in vivo.

Conclusions

- Long term SBS of metal bracket bonded to UTZ required both chemical and mechanical bond strengths.
- Roughening UTZ with dental burs produced less surface roughness and this method was free from airborne particles.
- Roughening UTZ with zirconia stone burs had acceptable long term SBS and resulted in easily to remove the remnant adhesive resin after bracket debonding.

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Declaration of Interest

The authors report no conflict of interest.

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