

THE INFLUENCE OF RESIN CEMENT THICKNESSES ON SHEAR BOND STRENGTH OF THE CEMENT-ZIRCONIA

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ABSTRACT

INTRODUCTION: Zirconia is a polycrystalline ceramic with no glassy phase like other glass ceramics. To gain a suitable retention, zirconia present some inherent problems. Surface treatment of etched zirconia with hydrofluoric acid does not adequately roughen the surface for the purpose of retention, while grinding zirconia to create surface roughness is often used as an option to improve its' mechanical bonding.

OBJECTIVES: The study examined the influence of resin cement thicknesses on shear bond strength of zirconia treated with a universal adhesive.

MATERIAL AND METHODS: Forty zirconia specimens were prepared, surface-treated with universal adhesive, and divided into 4 groups according to resin thicknesses (50, 80, 160, and 240 μm). All samples were stored in 37°C distilled water for 24 hours. Shear bond strength was performed using a universal testing machine at a 0.5 mm/min crosshead speed, until failure. Failure modes were analyzed using stereomicroscope at a magnification of $\times 50$. Results were statistically analyzed by one-way ANOVA and Tukey's multiple comparison tests.

RESULTS: The shear bond strength for each group was 30.28 ± 3.09 , 26.86 ± 2.21 , 25.98 ± 2.96 , and 18.22 ± 1.71 , respectively, and 50 μm significantly showed highest bond strength than in other groups ($p < 0.05$). There were no significant differences in bond strength between 80 μm and 160 μm ($p > 0.05$), while 240 μm significantly showed lowest bond strength than in other groups ($p < 0.05$). Adhesive failure was mostly found in all groups.

CONCLUSIONS: Resin cement thicknesses had influence on zirconia shear bond strength treated with a universal adhesive. The thinner resin cement showed higher shear bond strength than the thicker resin cement thicknesses.

KEY WORDS: zirconia, resin cement, universal adhesive, resin cement thickness.

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INTRODUCTION

Zirconia is a polycrystalline ceramic with no glassy phase like other glass ceramics. To gain a suitable retention, zirconia present some inherent problems. Surface treatment of the etched zirconia with hydrofluoric acid does not adequately roughen the surface for the purpose of retention, while grinding zirconia to create surface

roughness is often used as an option to improve its' mechanical bonding [1]. Retention of zirconia restoration can be achieved by both mechanical and chemical procedures. A well-recognized protocol for increasing mechanical retention is grit blasting or sandblasting [2]. Sandblasting is a technique, where aluminum oxide particles are released to attack the surface of zirconia, creating roughness on the surfaces. Surface irregularity

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is also known to increase surface energy and wettability of the zirconia [3]. Many studies had suggested that sandblasting can be a method that achieves mechanical retention in zirconia restoration, as zirconia cannot be etched by hydrofluoric acid in a normal condition [2, 4-6]. However, a chairside sandblasting device is a must for dental practitioner, and thus increasing cost and procedure time.

To further increase the longevity of bonded zirconia restoration, chemical retention has been recommended [2, 7]. Traditional bonding methods had been widely used and considered an effective bonding procedure in both zirconium restoration and the tooth abutment, in which the application of primer and bonding agents are required before cementation [2]. These primers and bonding agents contain essential functional monomer, which would bond to zirconia's surfaces. This traditional bonding requires multiple steps for surface treatment. Nowadays, a novel adhesive approach has been released into the market, which is known as 'universal adhesive' or 'multimode adhesive'. It aims to reduce clinical steps and errors [8]. The universal adhesive aims to incorporate potential chemical monomers into a single-bottle, which is designed to bond with both direct and indirect restorations, including ceramics, composites, and metals [9, 10]. This provides dentists with more options when choosing an optimal protocol for bonding with different prepared restorations. Many studies had reported that the use of universal adhesive can improve the bond strength of zirconia [10-12]. Kim *et al.* found that the universal adhesive exhibits significantly higher bond strength in comparison with the primers containing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) [13]. Santos *et al.* had shown that the use of universal adhesive may provide an adequate shear bond strength of zirconia for clinical use [10]. Moreover, many studies had claimed that universal adhesive may provide a sufficient shear bond strength on zirconia as an alternative option to traditional bonding [14-16].

Functional monomer that provides chemical retention for zirconia is typically 10-MDP, which is an acidic functional monomer containing phosphates. It is capable to create chemical bond with metal oxide surface [17]. Zirconia surface is covered with oxide layer, which makes it similar to metal surface, which has been successfully proven by various studies regarding application of 10-MDP to increase bond strength of zirconia [18]. However, the problem with 10-MDP is that it is susceptible to hydrolysis, which predisposes the interface to become prone to leakage [19]. Even so, 10-MDP is still an important aspect in most dental bonding agents [17, 18, 20].

Resin cements play an important role in filling up the internal gap between dental ceramic restoration and tooth abutment. Various dental resin cements are available on the market, providing options for luting

dental restorations. Resin cements contain various compositions according to different manufacturers in regard to modes of cure and types of surface treatment. These variations can differ dental cement properties, such as thickness, viscosity, and strength [21]. Grajower and Lewinstein found that the cement space of a crown should be set to be at least 50 μm , where the space of 30 μm would be reserved for the cement thickness and 20 μm would serve as a potential distortion during fabrication of restoration [22]. Taha *et al.* suggested that the use of 80 μm spacer thickness resulted in significantly higher retention than 100 μm and 120 μm in zirconia [23]. Currently, some researchers had investigated on the effect of resin cement thicknesses that affected mechanical properties of bonded restoration, such as bond strengths, fracture toughness, and color translucency [24]. A poor internal adaptation can lead to an increase in resin cement thickness, which may affect bond durability of the restoration [25]. This suggested that variations of cement thickness may have an effect on bond strength of zirconium restoration. Many experiments had been performed to measure the resin-zirconia bond strength under different conditions, including surface treatments, resin cements, and the use of primers with other factors [5, 10, 18, 26]. Until now, there has been no study that evaluated the resin-zirconia bond strength under conditions differing in the thicknesses of resin cement together with universal adhesives. Therefore, the null hypothesis of the present study was that the shear bond strengths of zirconia bonded with a universal adhesive with different resin cement thicknesses were not different.

OBJECTIVES

The purpose of the present study was to evaluate the influence of cement thicknesses on shear bond strengths of surface-treated zirconia with a universal adhesive.

MATERIAL AND METHODS

SPECIMEN PREPARATION

Forty fully sintered zirconia specimens (6.0 mm in diameter and 4.0 mm in thickness) were prepared from a zirconia block (Ceramill Zolid HT+PS, Amann Girschbach, AG, Koblach, Austria), and sintered according to the manufacturer's instruction. Specimens were embedded in polyvinyl chloride (PVC) tube, and filled with dental gypsum type IV. All specimens were polished with 600 grit silicon carbide paper (3M Wetordry abrasive sheet, 3M Minnesota, USA) with 2 kg/cm² force, 100 round/minute for 2 minutes under running water, using automatic polishing machine (Tegramin-25, Struers Inc., Cleveland, USA). Then, they were sandblasted (A10723

Base 3, Dental Vision Co. Ltd., Bangkok, Thailand) with 50 µm alumina for 15 seconds under 3.8 bar pressure and a 10 mm distance [27]. The specimens were rinsed off using running water and cleaned for 15 minutes in distilled water with ultrasonic cleaner (WUC-D22H, DAIHAN-brand Analog Ultrasonic Cleaners, DKSH Singapore Pte. Ltd., Singapore).

All specimens were surface-treated using a universal adhesive (Clearfill Universal bond and Clearfill DC activator, Kuraray Noritake Dental Inc., Tokyo, Japan) and divided into 4 groups with 10 specimens each, according to resin cement thicknesses: group 1: 50 µm, group 2: 80 µm, group 3: 160 µm, and group 4: 240 µm. Table 1 presents materials used in the present study.

A one-sided tape (PPM sticky tape, PPM industries, Brembate Sopra, Italy) with different thicknesses of 50, 80, 160, and 240 µm were cut with a dimension of 10 mm × 10 mm, and a hole was made with a diameter of 2 mm as bonded area. One side of the tape was cut until it reached the hole made to facilitate removal of the tape after bonding procedure. A one-sided tape was adapted on the top of zirconia surface.

ZIRCONIA SURFACE TREATMENT

A micro-brush soaked in an adhesive containing a mixture of 1 drop of clearfil universal bond and 1 drop of clearfil DC activator was applied on zirconia surface only 1 time, and new micro-brush was used to clean up the excess of adhesive inside one-sided tape. A water oil-free triple syringe was blown using a force of 40-50 pound/cm² with a distance of 10 mm, until the solvent in adhesive was totally dry. To evaporate the solvent from the zirconia surface, dry air was blown until there was no movement of the liquid and the shiny zirconia was presented. The samples were covered with a dark container to protect any contaminations before the next steps.

CEMENTATION PROCEDURE WITH RESIN CEMENT

The resin composite rods (CeramX spherotec 1 shade A3.5, Ivoclar vivadent AG, Schaan, Liechtenstein) were prepared using a silicone mold (Honigum putty, DMG

GmbH, Hamburg, Germany) of 3 mm in diameter and 2 mm in height. Resin composite rods were light-cured for 40 seconds using a light curing unit (Demi Plus, SDS Kerr, Middletin, WI, USA), which was held perpendicularly and closed to the sample as much as possible. Silicone mold was removed, and light curing was applied for another 40 seconds. Resin composite rods were polished using the same above-mentioned method. Resin cement (Multilink N, Ivoclar vivadent AG, Schaan, Liechtenstein) was mixed and injected into a hole made by one-sided tape area until it reached upper margin of the tape. Then, the excess resin cement was removed using a micro-brush. After that, a composite rod was placed onto resin cement with a force of 50 N using modified durometer, and the excess cement was further removed. The sample was light-cured on the top surface for 40 seconds, using the protocol mentioned above. Then, the one-sided tape was carefully removed. The samples were again light-cured for another 40 seconds at each side rotating at 90 degrees for each curing, until it reached 360 degrees. The specimens were left for another 10 minutes to ensure complete polymerization of the samples. Finally, the samples were incubated in distilled water at 37°C for 24 hours (Incubator; DI-150, Human Lab Inc., Gyeonggi-Do, Korea) before further analysis.

SHEAR BOND STRENGTH TEST

The adhesive area of each specimen was measured with a digital caliper (Digital Vernier Caliper Mitutoyo CD-6 CS, Mitutoyo Corp., Japan) before analysis of shear bond strength. The samples were tested for shear bond strength by a universal testing machine (AGS-X 500N, Shimadzu Corp., Kyoto, Japan). Each specimen was fixed in the testing machine, and a knife cutting-shaped shear blade was placed parallelly to the junction between the zirconia and resin cement. Crosshead was lowered until it came into contact with the specimen in pre-testing mode (Figure 1). Shear load was applied at a 0.5 mm/min crosshead speed, until failure. Debonded force was calculated in newtons (N); shear bond strength in was measured in megapascals (MPa) and was determined using a formula shear bond strength = F (force in N)/A

TABLE 1. Material composition used in this experiment

Material name	Compositions
Clearfill universal bond (Lot No. B10044)	Bisphenol A diglycidylmethacrylate, 2-hydroxyethyl methacrylate, ethanol, 10-methacryloyloxydecyl dihydrogen phosphate, hydrophilic aliphatic dimethacrylate, colloidal silica, dl-camphorquinone, silane coupling agent, accelerators, initiators, water
Clearfill dual cure activator (Lot No. CH0009)	Ethanol, catalyst, accelerator, sodium sulfinate
Multilink N automix (Lot No. X51463)	Bis-GMA, ethanol, 2-hydroxyethyl methacrylate, phosphonic acid acrylate, diphenyl(2,4,6-trimethylbenzoyl)phosphine oxide, potassium fluoride

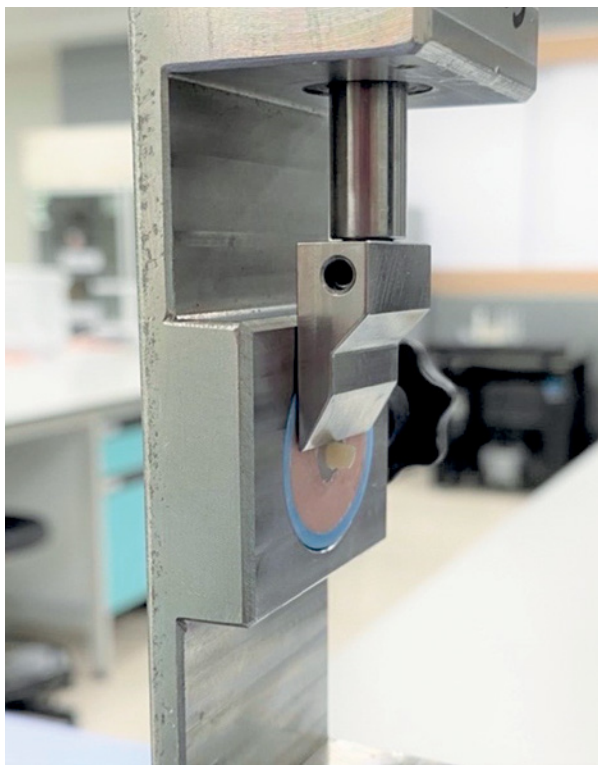


FIGURE 1. Configuration of shear bond strength test

(cross-sectional area of the resin cement-zirconia interface in mm²).

MODE OF FAILURE ANALYSIS

After testing of the shear bond strength, debonded surfaces were examined under a stereomicroscope (NexiusZoom, EVO, Euromex Microscopen B.V., Arnhem, the Netherlands) at a magnification of ×50 to evaluate the mode of failure consisted of 3 types: 1) an adhesive failure was the failure presented between zirconia and resin cement – this occurred when there was no resin cement remnant found on the zirconia surface; 2) a cohesive failure in resin cement was a failure that occurred within resin cement itself – this happened when there was a whole surface of resin cement found on the zirconia surface; 3) a mixed failure was a combination of adhesive failure and cohesive failure.

STATISTICAL ANALYSIS

Results of all groups were analyzed using SPSS version 20.0 software for Mac (SPSS Inc., Chicago, Illinois, USA) with 95% confidence level. Normality of distribu-

TABLE 2. Mean shear bond strength and percentage of failure mode

Groups	Mean shear bond strength (SD)	Percentage of failure mode		
		Adhesive	Mixed	Cohesive
1 (50 μm)	30.28 (3.09) ^a	90	10	0
2 (80 μm)	26.86 (2.21) ^b	70	30	0
3 (160 μm)	25.98 (2.96) ^b	70	30	0
4 (240 μm)	18.22 (1.71) ^c	50	50	0

The same letter indicates no statistically significance difference.

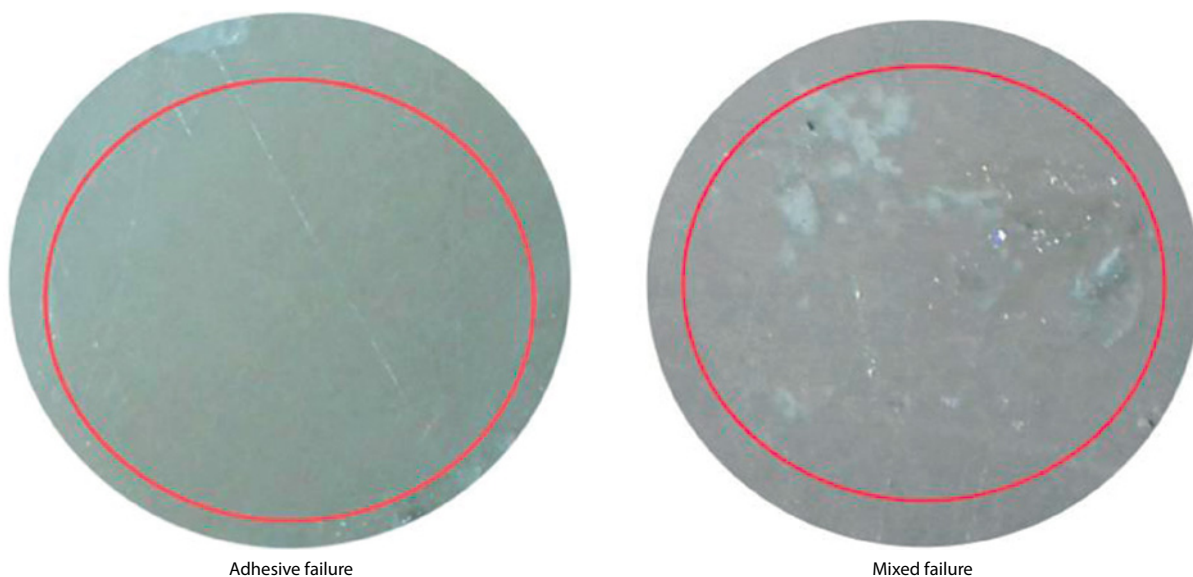


FIGURE 2. Illustrations from stereomicroscope demonstrate mode of failure

tion was analyzed with Kolmogorov-Smirnov test (KS test) and homogeneity of variance was examined with Levene's test. Bond strength values were further analyzed using one-way ANOVA to assess primary outcome, followed by Tukey's HSD test to assess multiple comparisons.

RESULTS

In the present study, the means and standard deviations of shear bond strength are shown in Table 2. Group 1 showed statistically highest shear bond strength among all cement thicknesses, while group 4 showed significantly lowest shear bond strength.

One-way ANOVA demonstrated that the shear bond strength in the cement thickness of 50 μm was significantly different from the cement thickness of 80, 160, and 240 μm ($p < 0.05$). Group 2 was significantly differed from group 1 and 4 ($p < 0.05$), while group 3 shear bond strength was not significantly different ($p > 0.05$). In group 3, the shear bond strength was significantly different from the cement thickness of groups 1 and 4 ($p < 0.05$). Group 4 shear bond strength was differed significantly from groups 1, 2, and 3 ($p < 0.05$).

The distributions of failure modes are summarized in Table 2. Group 1 showed 90% adhesive failure and 10% mixed failure. Groups 2 and 3 demonstrated 70% adhesive failure and 30% mixed failure. Group 4 showed 50% adhesive failure and 50% mixed failure. Figure 2 presents illustrations from stereomicroscope with mode of failure.

DISCUSSION

In the present study, the shear bond strength of zirconia bonding with universal adhesive in different resin cement thicknesses were significantly different. Therefore, the null hypothesis of the study was rejected.

Many studies had examined zirconia-resin bond strength with various surface treatments [5, 7, 11]. However, additional concern with zirconia is internal gap or cement space produced by CAD-CAM manufacturing technique, which might result in a larger resin cement space. The marginal and internal gap of zirconia restoration can range from 60 to 240 μm according to a study of Cunali *et al.*, which evaluated the space for resin cement during cementation [28]. In addition, Taha *et al.* study reported that 80 μm spacer thickness provided a significantly higher retention than 100 and 120 μm [23]. However, a study of Grajower and Lewinstein suggested that the cement space should be set at 50 μm where it would provide a 30 μm space for the area of cement and 20 μm for a potential distortion during material fabrication [22].

Thus far, there is no investigation on resin cement thicknesses influencing resin-zirconia bonding treated with a universal adhesive. For the first time, the present study investigated whether different resin cement thicknesses would affect resin-zirconia bonded with

a universal adhesive. Universal adhesive incorporates various functional monomers, which allow it to bond with various materials, including zirconia [8]. Moreover, universal adhesive shortens treatment time and may reduce contamination risk during bonding procedure. 10-MDP, one of the functional monomer, contains both phosphate and methacrylate groups, which allow these molecules to bond with both zirconia and resin [10, 17].

Therefore, our study conducted an experiment with various cement thicknesses of 50 μm , 80 μm , 160 μm , and 240 μm to evaluate whether 80 μm would still provide the highest shear bond strength when compared to the thinner resin cement thickness, as mentioned in a study by Taha *et al.* [23]. The 50 μm thickness represented the minimum cement space that should be set [22], whereas the 240 μm thickness provided the highest thickness that could be found in a previous study [28]. The 240 μm thickness was also chosen from previous studies since it was the highest range of cement gap that could be reproduced by stacking 80 μm tapes in the present study.

According to the results of the present study, group 1 significantly demonstrated the highest shear bond strength. The results from our study are in line with a study by Taha *et al.*, where the increase in cement thicknesses resulted in a lower shear bond strength. In our study, 80 μm resin cement thickness showed no significant differences from the 160 μm resin cement thickness, but it was significantly different from the thickness of 240 μm in shear bond strength. The reason for the increase in shear bond strength of thin resin cement may possibly be the lesser proportion of voids and defects produced in resin cement during cementation process. The void that may be presented in the resin cement could start crack propagation, which led to failure at lower forces. Group 4 was cemented with the thickest resin cement space, and this group of specimens presented the lowest shear bond strength. The mode of failure from the thinnest resin cement thickness (group 1) also demonstrated 90% adhesive failure and 10% mixed failure. It was observed that the more mixed failure occurred in the thicker resin cement, the more chance of voids found in the resin cement. The void presented in the resin cement could create a stress concentration when force is applied, which can lead to crack initiation and eventually, increase change in failure from adhesive to mixed failure. This could be the reason why most mixed failures occurred in group 4 compared to other groups. However, a mixed failure cannot be considered the true failure. Furthermore, a finite element experiment by Liu *et al.* reported that higher stress can be developed within the resin cement when the thickness increased from 10 to 180 μm [29]. Further studies should investigate different resin cement thicknesses of zirconia treated with different bonding agents, and evaluate bond durability of zirconia and resin cement interface by a thermocycling method.

CONCLUSIONS

Within the limitation of the present study, it can be concluded that the resin cement thicknesses had an influence on zirconia shear bond strength.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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