Shear bond strength of Zirconia to different adhesive resin cements

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Abstract

Objectives: This in-vitro study investigated the shear bond strengths (SBS) and failure modes of 2 resin cements bonded to zirconia core ceramic.

Materials and methods: Forty square-shaped (8 mm x 8 mm x 2 mm) and cylinder-shaped (5 mm diameter and 3 mm height) zirconia blocks were prepared and randomly assigned into two groups (n=20). In Group 1, the square-shaped zirconia specimens were bonded to the cylindrical zirconia using RelyX™ Ultimate in combination with Scotchbond™ Universal adhesive. In Group 2, the bonding of zirconia specimens was made using Panavia™ F2.0. Punched adhesive tape with 5 mm diameter hole was placed on the square- shaped zirconia blocks to restrict the bonding surface area. Before bonding, the surfaces of the square and cylinder-shaped blocks were sandblasted with 50 µm Al_2O_3 at 2-bar pressure. After bonding, specimens were stored in distilled water at 37 °C for 24 hrs and then shear bond test was performed using a universal testing machine. The bond strength data were analyzed using an independent t-test at α = 0.05.

Results: There was no significant difference between shear bond strength of either RelyX™ Ultimate (17.2 ± 6.0 MPa) or Panavia™ F 2.0 (15.8 ± 4.4 MPa) bonded to zirconia. The failure in all specimens originated at the interface between resin cement and zirconia.

Conclusion: The shear bond strength of RelyX™ Ultimate was comparable to the group bonded to Panavia™ F 2.0 bonded to zirconia. The failure mode of all specimens was interfacial failure.

Key words: shear bond strength (sbs), zirconia, adhesive , resin cement, phosphate monomer, interfacial failure

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Introduction

Zirconia is a polycrystalline ceramic utilized in fabrication of fixed partial dentures. It gains rapid popularity due to its superior fracture toughness, excellent chemical stability and natural-look appearance.[1-2]

Many surface treatment concepts for bonding of polycrystalline ceramic have been proposed in the literatures such as grinding, airborne particle-abrasion, silica surface coating, laser treatment, acid etching and primer application.[3-8] Hydrofluoric acid which has capability to etch silicon dioxide is used as a surface treatment on most glass-based dental ceramics before applying a resin cement to increase micromechanical retention and surface bonding area. However, polycrystalline ceramics such as aluminium or zirconium dioxide have very limited amount of silicon dioxide, thus hydrofluoric acid etching has less or no significant effect to increase surface irregularity and surface bonding area.[1,9-10] Hydrofluoric acid etching followed by silane coupling agent is an unpredictable method to bond zirconia to resin cements.[11] Surface coating and laser treatment (Er:YAG, Nd:YAG and CO_2 laser) usually need additional equipments which are not practical in day-to-day practice.[8] As-produced and grinding or polishing surface treatment provide the least bond strength compare to other surface treatments.[12-13] Airborne particle-abrasion with Al_2O_3 particles could increase the bonding surface area and bond strength.[14-15]

Many resin cements have been improved in order to increase the bond strength to zirconia. Resin cements containing phosphate monomer (MDP, 10 methacryloyloxydecyl dihydrogen phosphate) were reported to provide a strong and long lasting resin-zirconia bond.[16-18] MDP is a functional group with a long organic hydrophobic chain molecule with two ends. One end has vinyl groups that react with the monomers of the resin cement when polymerized. At the other end, hydrophilic phosphate ester groups bond strongly with metal oxides, or calcium hydroxyapatite.[19] MDP is a functional component for some dual-cure resin cements such as Panavia™ F 2.0 and bonding agents such as Scotchbond™ Universal adhesive. Wolfart .et al. reported that mechanical surface roughening on zirconia surface by air abrasion with Al_2O_3 particles prior to adhesive bonding and the use of MDP-containing resin cements provided superior and long lasting bond strength.[16] However, there is no bonding concept can offer strong and reliable long-term bonding adhesive resin cements and zirconia in oral function.[20]

The bond strength of dental materials can be assessed by variety of methods such as shear, micro-shear, tensile and micro-tensile testing techniques.[21-22] In order to determine the strength at the interface, shear and tensile bond strength tests have been performed exclusively in relatively large bonding surface area of dental specimens. Tensile bond test provides lower mean bond strength value, low standard deviation (homogeneous data value) and more uniform stress distribution.[23-24] However, shear bond strength test is considered more clinically relevant because resistance to shear stresses is similar to the force exerted to retain the restorations that are bonded to tooth structure or core materials.[25] The shear bond strength can be calculated by Equation 1.

$$R = F/A \qquad (1)$$

Where "*R*" is strength (MPa), "*F*" is load at fracture (N) and "*A*" is the interfacial bonding area of the specimen (mm^2).

The purpose of this study was to compare the shear bond strengths of RelyX™ Ultimate and Panavia™ F 2.0 resin cements bonded to zirconia (Lava™ Plus, 3M ESPE). The failure modes of bonded specimens were also determined.

Materials and methods

The details and composition of materials used in this study are shown in Table 1

Specimen preparation

Pre-sintered zirconia blocks (Lava™ Plus ; 3M ESPE, USA) were prepared into forty square and forty cylinder–shaped blocks. Square-shaped blocks were 10 mm x 10 mm x 2.5 mm. Cylinder-shaped blocks were 6.25 mm in diameter and 3.75 mm in height in pre-sintered stage. Before sintering, the bonding surfaces were polished using 1,200 grits silicon carbide paper to standardize the bonding surface, then fully-sintered following the manufacturer's instruction using a ceramic furnace (Lava™ Furnace 200, 3M ESPE,USA) at a temperature between 1,350 and 1,500 °c. After fully-sintered, the final dimension was approximately 8 mm x 8 mm x 2 mm for the square specimens and diameter of 5 mm and 3 mm in height for cylindrical block (Fig.1). The fully-sintered square-shaped zirconia blocks were embedded in self-cured acrylic resin (Quick Resin, Shofu, Japan), in PVC tube (26 mm of diameter and 15 mm in height) by exposing one surface for bonding (Fig.2). The mean diameter of cylinder-shaped zirconia blocks of group1 and 2 were 5.03 ± 0.11 mm and 5.05 ± 0.08 mm respectively.

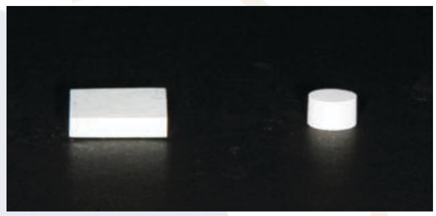

Fig. 1 Pre-sintered of square and cylinder-shaped zirconia specimens

Table 1 Composition of materials used in this study

Material/Trade name	Composition	Manufacturer
Lava™ Plus Zirconia	NA	3M EPSE, USA
RelyX™Ultimate Dual cure adhesive resin cement	1. Base Paste - Methacrylate monomers, Radiopaque silanated fillers, Initiator components, Stabilizers, Theological additives 2. Catalyst Paste - Methacrylate monomers, Radiopaque alkaline fillers, Theological additives, Fluorescence dye	3M EPSE, USA
Scotchbond™ Universal Adhesive Resin cement	- MDP Phosphate monomers, Dimethacrylate resins, HEMA, Vitrebond Copolymer, Filler, Ethanol	3M EPSE, USA
Panavia™ F 2.0 Dual cure adhesive resin cement	1. A Paste - 10-Methacryloyloxydecyl dihydrogen phosphate (MDP), Hydrophobic and hydrophilic dimethacrylate, Silanated silica filler and colloid, Camphorquinone 2. B Paste - Hydrophobic and hydrophilic dimethacrylate, Silanated barium glass filler, Surface treated sodium fluoride	Kuraray, Japan

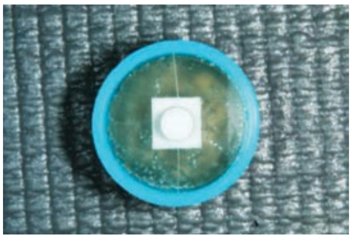

Fig. 2 Specimen after bonding and ready for incubation

The bonding surface of square and cylinder-shaped specimens were sandblasted using aluminum oxide particles with grain size of 50 μm at the distance of 10 mm perpendicular to the bonding surface at pressure of 2 bars for 15 seconds (Sandblasted machine, Vario Basic® Renfert, Germany). The sandblasted zirconia blocks were cleaned with distilled water in ultrasonic bath for 10 minutes, pressure-steamed and dried by oil-free air compressor. Punched adhesive tape (Magic™ adhesive tape, 3M ESPE, USA) with a diameter of 5 mm were placed on the square-shaped zirconia block to restrict the bonding surface area. The fully-sintered cylindrical zirconia blocks were bonded to the square-shaped zirconia over the punched adhesive tape using two different types of resin cement.

In group 1 (n=20) : The bonding surface of both square and cylindrical zirconia blocks was coated with Scotchbond™ Universal Adhesive for 20 seconds then air dried. RelyX™ Ultimate was mixed following the manufacturer's instructions and applied to both the square and cylinder-shaped zirconia surfaces. The cylinder-shaped zirconia was attached immediately to square-shaped block and loaded on the top of cylinder-shaped zirconia using a 1 kg load. The excess resin cement was removed immediately after loading by using disposable micro-brush, light-cured for 20 seconds on four different locations and oxyguard was applied. The bonded specimens were further kept under load for 10 minutes. The adhesive tape was finally removed to avoid any force to the bonding interface.

In group 2(n=20): Panavia™ F 2.0 was mixed following the manufacture's instructions and then applied to both the square and cylinder-shaped zirconia surfaces without any ceramic primer. The cylinder-shaped zirconia was attached immediately to square-shaped block and loaded on the top of cylinder-shaped zirconia using a 1 kg load. The excess resin cement was removed immediately after loading by using disposable micro-brush. The oxyguard was applied. The bonded specimens were kept under load for 10 minutes.

Testing procedure

All specimens were stored in distilled water at 37 °C immediately after bonding for 24 hours before testing. The specimens were attached to the testing jig in a universal testing machine (Fig. 3), (Model 5566, Instron Ltd, Coronation Road High Wycombe, Buckinghamshire, England) for a shear bond test. The shear chisel loading device (Fig.3) was loaded parallel to the bonding surface at a distance of 0.5 mm away from the interface. The crosshead speed was 0.5 mm/minute. The failure loads were recorded in Newton. The

Fig. 3 Shear bond strength testing assembly

shear bond strengths in MPa were calculated by Equation 1.

The debonded surface were examined under scanning electron microscope (Jsm-6610lv, Jeol Ltd, Tokyo, Japan). The failure mode was classified into two types based on the origin of failure.

- Adhesive failure or interfacial failure: the failure originated between the resin cement and zirconia interfaces
- Cohesive failure: the failure originated within the bonding material (resin cement) or in the zirconia

Statistical analysis

Data were recorded in MPa for shear bond strength and analyzed using an independent t-test (α = 0.05).

Results

The mean shear bond strength values of zirconia to two resin cements are summarized in Table 2.

There was no significant difference between SBS of zirconia bonded to RelyX™ Ultimate and those bonded to Panavia™ F 2.0.

From the loading area (Fig.4), The failure in all specimens of both groups was originated at the interface between resin cement and zirconia. It appeared that during testing, the resin cement peeled off from the zirconia surface and propagated into the resin layer. There was no zirconia fracture observed in this study.

Table 2 Shear bond strength of both groups

Group	Mean shear Bond Strength (MPa)	Mode of failure
1	17.23 ± 6.03	interfacial
2	15.80 ± 4.46	interfacial

1= RelyX™ Ultimate bonded to zirconia
2= Panavia™ F 2.0 bonded to zirconia

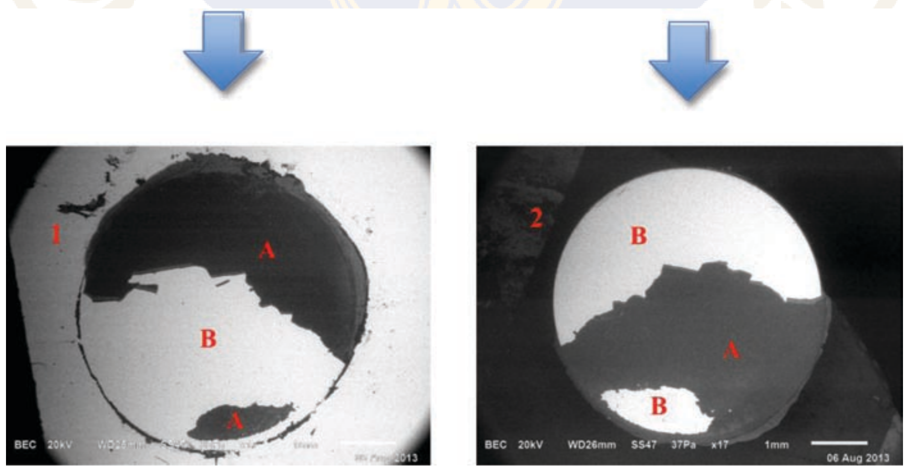

Fig. 4-1 The representative SEM photographs of fractured surfaces of a specimen in group 1 at magnification of 17x: (1) square-shaped zirconia block and (2) cylinder- shaped zirconia block, (A): resin cement, (B): Zirconia. The arrows show the loading area during shear bond test.

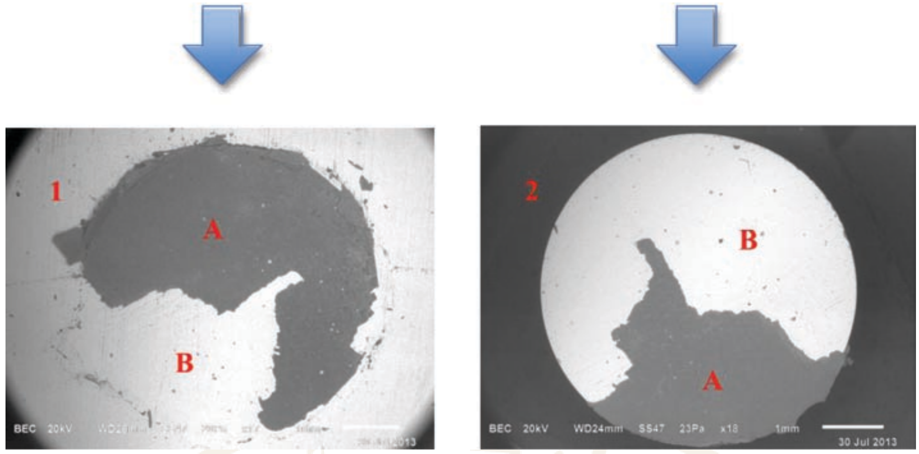

Fig. 4-2 The representative SEM photographs of fractured surfaces of a specimen in group 2 at magnification of 17x: (1) square-shaped zirconia block and (2) cylinder- shaped zirconia block, (A): resin cement, (B): Zirconia. The arrows show the loading area during shear bond test.

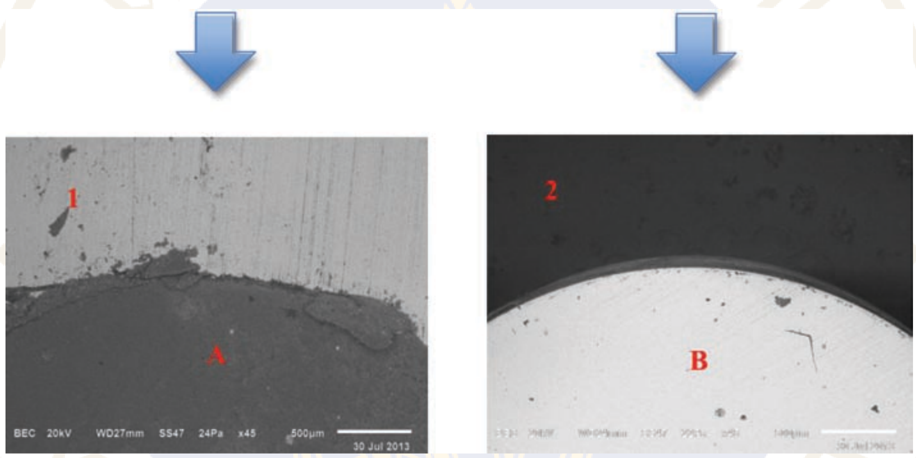

Fig. 4-3 The representative SEM photographs at origin of failure of a specimen in group 1 at magnification of 45x: (1) Square-shaped zirconia block and (2) cylinder- shaped zirconia block, (A): resin cement, (B): Zirconia. The arrows show the loading area during shear bond test.

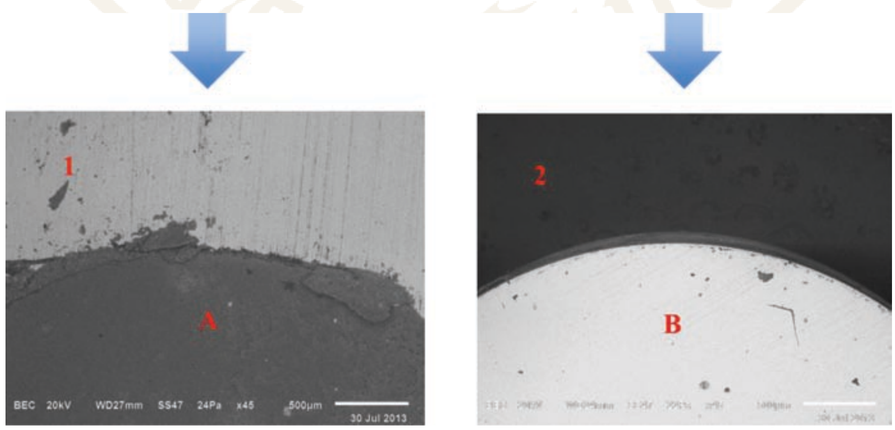

Fig. 4-4 The representative SEM photographs at origin of failure of a specimen in group 2 at magnification of 45x: (1) Square-shaped zirconia block and (2) cylinder-shaped zirconia block, (A): resin cement, (B): Zirconia. The arrows show the loading area during shear bond test.

Discussion

There was no significant difference between SBS of zirconia (Lava™ Plus ESPE) bonded to RelyX™ Ultimate and those boned to Panavia™ F 2.0 with the bond strength of 17.23 ± 6.03 MPa and 15.80 ± 4.46 MPa, respectively. The main reason might be the similarity in compositions of both resin cements, especially the functional monomer (MDP), which was reported to be able to bond to zirconia.[16,20,26] Additionally, The mean SBS values were similar to results obtained from a previous study with the mean SBS ranking from 12.5 MPa to 17.1 MPa.[12] Akin H. et al. reported that the means SBS of different surface- treated zirconia bonded to Variolink II (Ivoclar Vivadent, Liechtenstein) ranking from 1.5 MPa to 4.7 MPa.[8] Some previous studies reported that the means SBS of zirconia bonded to different MDP-containing resin cements were approximately 30.5 –53.8 MPa which were higher than the means SBS obtained in this present study.[20, 27-29] The discrepancy of means SBS between these studies might be due to the use of different testing methods, testing conditions, pretreatment surface of bonded zirconia, different adhesive resin cements and specimen preparation.

The mean SBS of zirconia bonded to RelyX™ Ultimate was slightly higher than those bonded to Panavia™ F 2.0. Panavia™ F 2.0 was used to bond to zirconia without any primer, especially ED primer. Main compositions of ED primer are HEMA and MDP. ED primer was not used in this study, because ED primer is a dentin bonding agent. Therefore it is not suggested to use with any kinds of ceramic restoration. ED primer is an initiator of self-cured mechanism, thus without this primer the dual-cured adhesive resin cement might not be able to cure completely by light-cured. RelyX™ Ultimate adhesive resin cement was used with Scotchbond™Universal Adhesive. Scotchbond™Universal Adhesive contains MDP functional monomer, HEMA, silane coupling agent and initiators (Table 1). Scotchbond™Universal Adhesive is recommended to use on both tooth structure and ceramic restoration. Thus, it might promote better surface wettability. In a previous study, Magne P. et al. reported that silane coupling agent could improve bond strength of zirconia by increased wettability.[30] From fracture surface observation using SEM (Fig. 4), the specimens failed at the interfacial surface between resin cement and zirconia. Resin cement was peeled off from sandblasted zirconia surface, thus it might be questionable regarding the occurrence of chemical bond between MDP-containing resin cement and zirconia surface. However, the bond strength of zirconia to MDP- containing resin cement was possibly improved by clean and rough surface, surface wettability and low viscosity of adhesive resin cements.[29,31] In this study, Bonding surfaces of zirconia were sandblasted using Al2O3 particle with grain size of 50 µm at 2-bar pressure. This surface pretreatment could provide durable resin-to-zirconia bonds as reported in many studies.[4, 31, 32]

Blatz M. et al. conducted an in vitro study to evaluate and compare shear bond strengths of resin to densely sintered high-purity zirconia.[33] Composite cylinder-shaped blocks (3 mm diameter and 2.9 mm height) were used to bond to sandblasted zirconia (10 mm x 10 mm x 2 mm). They reported that cohesive failure was observed from 9-91%. In another study, cylinder-shaped zirconia blocks were used to bond to dentin using adhesive resin cements. The failure mode was observed under SEM and classified according to the existing of materials on the fracture surface. Akin H. et al. reported that the failure mode was 69% adhesive failure, 2.5% cohesive failure and 28.5% mixed failure.[8] However, There were two different adhesive interfaces, dentin- resin cement and zirconia-resin cement interface, so the details of adhesive and cohesive failures were not clearly reported. One of these two interfaces might occur separately. If the interfacial failure occurred at zirconia-resin interface, this would reflect the bond strength of zirconia to adhesive resin cement. If the interfacial failure occurred at the dentin-resin interface, it would reflect the bond strength of dentin to adhesive resin cement. The conclusion might be different if the failure mode was classified. The causes of cohesive failure occurred in shear bond test might be an inadequate strength of material such as composite or dentin and non-uniform stress distribution along the interface.[34] During shear bond test the stress might concentrate on composite or dentin than on the bonding interface, thus caused the failure to occur within the material.

According to the results from previous studies[8,34], two different interfaces were considered when bonded ceramic to dental substrates. Dentin bonding agent is used to bond tooth structure or composite blocks. Resin cement is used to bond zirconia. The bonding of these two interfaces should be optimized because the performance of bonded restorations would be determined by the weaker one. It has been stated that bond strength values obtained from a fractured surface containing cohesive failure of dentin or resin block would be meaningless as the measured "nominal" bond strength would reflect a mixture of mechanical properties of dentin and/or resin rather than the performance of the adhesive testing.[22,34-36] Percentage has been used to describe the failure mode, which classified as "cohesive" when the fracture surface occurred in dentin, or resin, "adhesive" when the fracture occurred at the adhesive interface, and "mixed" was considered as a mixture of adhesive and cohesive failure within the same fracture surfaces. In the "mixed" failure mode, the crack path might remain mainly within the adhesive interface but also included some small areas of the resin and/or dentin, or if a larger region of the fractured surface also included cohesive failure in dentin and/ or resin. The latter should clearly be rejected from the data because it would reflect about fracture strength of dentin or resin rather than bond strength.[22,35,37]

This present study was designed to restrict the failure to occur at the interface between zirconia to resin cements. This design could better evaluate the bond strength of zirconia to resin cement. The failure mode was classified by initial crack whether originated from the resin cement (cohesive failure) or at interfacial surfaces (adhesive failure). Both of bonding surfaces (square and cylinder-shaped) were fully-sintered zirconia, which had superior strength compared to zirconia-resin interface, thus cohesive failure of zirconia was eliminated. Moreover, crack initiation was concentrated at the bonding interface. The result revealed that the fracture of tested specimens originated from the zirconia- resin interface, which reflected the true "nominal" bond strength of zirconia-resin interface.

The limitation of this study was that the design was based on the use of only two dual-cured adhesive resin cements which contain MDP functional monomer and could not be implied to other luting cements. Other luting cements and long term simulated oral conditions should be considered in future research to further evaluate the durability and bond strength of zirconia bonded to adhesive resin cement.

In conclusion: within the limitation of this study, the SBS of zirconia bonded to RelyX™ Ultimate was not significantly different to those bonded to Panavia™ F 2.0 at 24 hours. the failure mode of all specimens in both groups was interfacial.

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