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Effect of a glaze layer on adhesion energy between resin cements to zirconia ceramic

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ABSTRACT

Purpose: To evaluate the effect of an etched and silanized glazed porcelain layer on the interfacial fracture toughness between a zirconia ceramic and resin cements.

Materials and methods: Forty rectangular-shaped yttrium stabilised zirconia ceramic plates were sintered and sandblasted with 100 μm Al_2O_3. Twenty specimens were glazed with Akzent Glaze Spray and then etched with 9% hydrofluoric acid for 3 min prior to being silanized with Monobond-S. Glazed and non-glazed specimens were further divided into two groups (n = 10) and allocated to one of two resin bonding systems, Variolink II and Multilink-Automix (Ivoclar Vivadent). The Multilink-Automix groups were treated with Metal/Zirconia Primer. Glass rods (12 mm) were bonded to each prepared zirconia plate, using the two bonding systems and were loaded to failure using a universal testing machine. Strain energy release rate (bond) values were calculated and de-bonded specimens were examined using SEM to determine the modes of failure. Data were analysed using two-tailed T-test and Dunnett-T3 post-hoc tests with statistical significance set at $p < 0.05$.

Results: Glazed zirconia surface significantly improved the mean bond values in the Variolink II group ($p < 0.05$), with no significant change ($p > 0.05$) in the Multilink-Automix Metal/Zirconia Primer group; the Multilink – Automix group, produced significantly higher bond values in the non-glazed group ($p < 0.05$). Fractographic analysis showed predominantly cohesive failure for the glazed groups and adhesive failure in the non-glazed groups.

Conclusion: Glazed zirconia surfaces produced higher resin cement bond strength than unglazed zirconia, however the combination of silanised phosphate monomer and metal/zirconia primer produced weaker bond on glazed zirconia.

1. Introduction

Full ceramic dental restorations have gained increasing popularity in the last two decades [1,2]. 3 mol% yttria- stabilized zirconia polycrystal (YTZP) is currently considered to be the strongest and most durable ceramic available in dentistry [3–6]. The excellent physical and mechanical properties of zirconia makes it an ideal and versatile restorative dental material [6,7]. However, due to the chemical inertness of zirconia, bonding this material to abutment teeth is challenging [8]. Unlike zirconia, silica based ceramics have micro-structural components that can be removed by an etching process, and chemically activated with silane to promote adhesion of the ceramic to the tooth structure [9–14]. The current recommended regime for bonding zirconia to the remaining tooth structures incorporates the use of particle air-abrasion (commonly known as sandblasting) and the use of a resin bonding system with MDP monomer coupling agents or phosphorylated methacrylate; this has been substantiated by high clinical success rates [15–18]. Other reported methods for improving resin bond strength to zirconia includes selective infiltration etching [19–21], hexamethylbisiloxane deposition with plasma spraying [22], surface fluorination [23], chlorosilane vapor phase pre-treatment [24], pyro-silicoating [25]; tribo-chemical silica coating [26–28] and laser irradiation [29–31]. Some studies have used the shear bond strength method and commonly used adhesive systems to investigate the influence of a thin layer of ceramic glaze fused to a zirconia substructure, as opposed to unglazed zirconia [22,32–37]. Unfortunately, shear bond strength and other methodologies that measure the critical stress at failure have inherent limitations, mainly that these are only capable of measuring the comparative stresses upon catastrophic failure [38]. It has been reported that these values do not represent the actual energy or work

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required to separate two dissimilar material interfaces, in addition to being drastically influenced by small deviations in sample preparations, making valid comparison across studies problematic [39]. A more appropriate methodology is to incorporate a fracture mechanics approach, which has already been shown to be a reliable method to evaluate the bonding characteristics in porcelain-fused to metal (PFM) and all-ceramic systems [40–42]. This approach has yet to be applied to resin cement based bonding systems. Another advantage to using a fracture mechanics approach is that it would be able to provide a better intrinsic estimation of the interfacial properties during crack propagation.

The purpose of this study was to determine the interfacial fracture toughness between glazed zirconia and two different resin bonding systems, using a fracture energy release approach [43].

2. Materials and methods

Forty rectangular (20 mm × 7 mm × 2 mm) zirconia plates (VITA In-Ceram, VITA Zahnfabrik; Germany) were sectioned under irrigation using a diamond grit blade on a low speed sectioning machine (Acutone; Struers, Denmark). The plates were planed under irrigation using 1000-grit silicon carbide abrasive paper and sintered according to manufacturer instructions. The sintered plates were sandblasted at 2 bar pressure with 100 µm aluminum oxide (Al_2O_3) at a distance of 10 mm until a homogenous surface texture was achieved. All specimens were ultrasonically cleaned in an acetone bath for 6 min and dried with clean compressed air. The specimens were divided into two groups (n = 20). One group was coated with three thin coats of VITA Akzent Glaze Spray (VITA Zahnfabrik) at a distance of 10 cm and fired in a ceramic furnace (Programat P300; Ivoclar Vivadent) according to the manufacturer's instructions. The glazed specimens were then etched for 180 s with 9% hydrofluoric (HF) acid (Ultradent Products;, UT, USA), thoroughly rinsed with running tap water, steam-cleaned (Steamer X3; Amann Girrbach, Austria) for 1 min and dried with clean compressed air. Monobond-S (Ivoclar Vivadent) was applied to the etched surfaces and dried for 1 min with clean compressed air. The remaining 20 plates remained sandblasted only with no further surface treatments (Fig. 1). A chevron shaped bond surface was created on all the zirconia plates by applying a custom cut-out sticker made of ± 50 µm thick non-stick polymeric transparent PVC film (Grafiprint, Belgium). Forty borosilicate glass rods (4 mm diameter × 15 mm) (PYREX Brand; Pt. Iwaki Glass, Indonesia) ends were then ground flat using 200 grit diamond belt, ultrasonically cleaned in acetone for three minutes, then dried with clean compressed air. The ground glass rod surfaces were etched with 9% HF acid (Ultradent Products) for 180 s, followed by rinsing with tap water for 1 min, steam cleaned and air dried, then silanized with Monobond-S following manufacturer's instructions. Variolink II base paste (Ivoclar Vivadent) cement was applied to the exposed chevron surface of 10 glazed and 10 sandblasted zirconia plates with a micro brush. Glass rods were bonded to the prepared zirconia plates by carefully positioning the silanized glass rod surface over the cement coated area with minimal pressure applied to ensure even coating of the cement on the adhesive area. The resin cement was then light-cured for one minute (15 s per quadrant) using an LED photo-polymerization unit (Bluephase 20i; Ivoclar Vivadent) with a light intensity of 1200 mW/cm^2 and working distance of 2 mm. The remaining glazed zirconia (n = 10) and sandblasted zirconia (n = 10) plates, received a layer of a phosphate monomer primer (Metal/Zirconia Primer; Ivoclar Vivadent). This was applied for 180 s followed by air-drying. Multilink-Automix base and catalyst pastes were dispensed at 1:1 ratio on a pad and hand-mixed for 10 s, after which it was applied to the chevron-shaped adhesive surfaces of all the remaining zirconia plates. The remaining etched glass rods were cemented to the zirconia plates in a similar manner as the Variolink II groups (Fig. 2). All prepared specimens were kept in a water bath at 37 °C for 24 . The test groups are summarized in Fig. 1 with the materials used listed in Table 1.

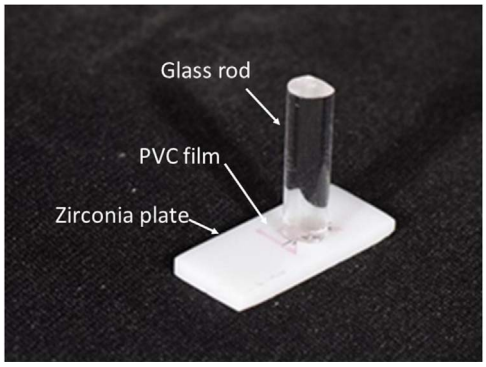

Fig. 2. Etched and silanized glass rod cemented to the zirconia plate with the non-stick PVC film in place, creating the chevron-shaped adhesive area.

2.1. Interfacial fracture toughness test

The specimens were clamped on a custom made jig to ensure correct orientation and to minimise compliance, then loaded 10 mm from the bonded interface, using a rounded steel pin with a diameter of 5 mm (Fig. 3). A universal testing machine (Instron, model 3369, Instron Corp., MA, USA) was used to apply a constant load, using a 50 N load cell and cross-head speed of 0.5 mm/min, until failure occurred. The load at failure was recorded using Istron Bluehill 3 software (Instron Corp.).

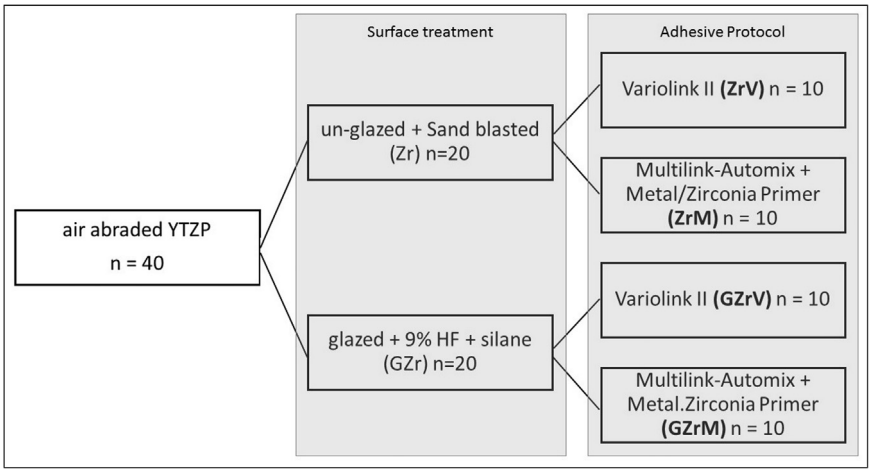

Fig. 1. Schematic flow-diagram of the experimental design including the applied surface treatment followed by the adhesive protocol. The final test groups are indicated in bold.

Table 1
Summary of materials used in the study.

Trade name	Chemical composition	Batch no.	Manufacturer
Monobond-S	3-methacryloxypropyltrimethoxysilane, ethyl alcohol and water	S39061	Ivoclar Vivadent; Schaan, Liechtenstein
Metal/Zirconia Primer	Solvent, phosphonic acid acrylate, ethoxylated Bis-EMA, initiators and stabilisers	T28760	Ivoclar Vivadent; Schaan, Liechtenstein
Variolink II	Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate, barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass and spheroid mixed oxide	T04188	Ivoclar Vivadent; Schaan, Liechtenstein
Multilink-Automix	Dimethacrylate, hydroxyethyl methacrylate (HEMA), barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass and spheroid mixed oxide	T22247	Ivoclar Vivadent; Schaan, Liechtenstein
VITA In-Ceram YZ	ZrO_2 91–94%, Y_2O_3 4–6%, HfO_2 2–4%, Al_2O_3 < 0.1%, SiO_2 < 0.1% and Na_2O < 0.1%		VITA Zahnfabrik; Bad Sackingen, Germany
VITA Akzent glaze spray	Not available	17620	VITA Zahnfabrik; Bad Sackingen, Germany
Ultradent Porcelain Etch	9% buffered hydrofluoric acid	B91TZ	Ultradent Products; South Jordan, UT, USA
PYREX brand borosilicate glass rod	SiO_2 80.9%, Al_2O_3 2.3%, Fe_2O_3 0.03%, B_2O_3 12.7%, Na_2O 4.03% and K_2O 0.04%		PT. Iwaki Glass; Indonesia

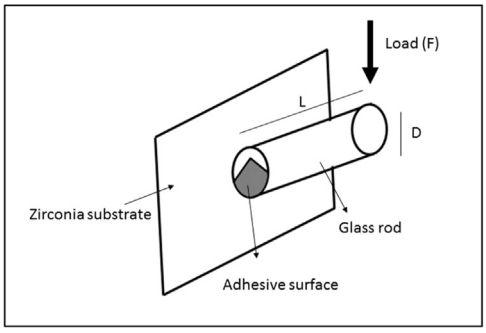

Fig. 3. A schematic illustration of a bonded specimens in load configuration. This diagram was adapted from Cheng et al. [43].

Interfacial fracture toughness values were calculated using the following formula [43]:

$$G_{IC}(J/m^2) = \frac{104.5\ F^2L^3}{ED^6}$$

where:

F = load at failure (N)
L = distance from bonded interface to loading point
E = elastic modulus of glass rod (64 GPa)
D = diameter of glass rod

Statistical analysis was performed using SPSS Statistics 22.0 software for Windows (IBM; NY, USA) with a statistical significance set at $p < 0.05$. Strain energy release rate data were analyzed using one-way ANOVA followed by multiple comparison between the groups using Dunnett-T3 test. The interaction between the use of glaze and type of cement were analyzed using two-way ANOVA.

2.2. Failure analysis

The fractured surfaces of the bonded glass rods and zirconia substrates were examined with a light stereoscopic microscope (SMZ745T, Nikon, Tokyo, Japan) to determine the mode of failure for each specimen. The glass rods were mounted with the fractured surface facing up on a custom-made jig aligning the rod perpendicular to the microscope mounting platform. The modes of failure investigated were allocated to one of the following 3 classification: adhesive (failure between the resin and zirconia substrate interface), predominantly cohesive (failure within the resin in which resin can be observed on both glass rod and zirconia substrate), mixed mode (adhesive and cohesive). The fractured specimens were then mounted and sputter coated in an Emitech SEM coating unit (Quorum Technologies Ltd, Kent, England) with 10 nm layer gold-palladium alloy and, analysed under scanning electron microscopy (JSM 6700 FE-SEM, JEOL).

Table 2
Results for the plane strain energy release rate for etched glass rods bonded to sandblasted zirconia vs. glazed and etched zirconia. The asterisk indicates significant increase in the bond energy.

Zirconia surface treatment	Bond system	Group	n	Mean (J/m^2)	Standard deviation
Glazed, etched and silanisation **(GZr)**	Variolink II	GZrV	10	202.35*	54.93
	Multilink-Automix and Zirconia/Metal Primer	GZrM	10	68.51	26.11
Non-glazed **(Zr)**	Variolink II	ZrV	10	19.04	6.56
	Multilink-Automix and Zirconia/Metal Primer	ZrM	10	59.40*	28.24

3. Results

The mean strain energy release rates for the groups are presented in Table 2. Glazed zirconia followed by hydrofluoric acid etching and silanization (GZrV) significantly improved the interfacial strain energy release rate of the Variolink II resin cement (ZrV) (ANOVA, $p < 0.05$). However, no significant improvement in critical plain strain energy release values could be established in the glazed/Multilink-Automix group (GZrM) when compared with the non-glazed group (ZrM) (T3 Test, $p > 0.05$). In the case of the phosphate- based Multilink-Automix resin in combination with Metal/Zirconia Primer (ZrM), significantly higher strain energy release rates were observed compared with the non-phosphate based resin Variolink II (ZrV) (T3 Test, $p < 0.05$).

The classifications of the failure modes are predominantly cohesive failure (fracture within resin), mixed failure (fracture within resin and adhesive failure between resin and zirconia) and adhesive failure (fracture between zirconia and resin interface). The percentages for the modes of failure for each group are presented in Fig. 4. Failure analysis with light microscope and SEM showed no adhesive failure at the resin-glass rod or zirconia-glaze interfaces. Adhesive failure at the resin-zirconia interface was found exclusively in the group with the lowest GIC (ZrV) (Fig. 5A), while group GZrV (Fig. 5B) predominantly failed cohesively within resin cement. Lastly, mixed mode failures and adhesive failure were found in both the ZrM (Fig. 5C) and GZrM (Fig. 5D) groups.

Examination of groups GZrM and ZrM using SEM revealed the presence of air bubbles in the resin cement. In group GZrM, a transitional zone between the zirconia and resin interface was observed at the

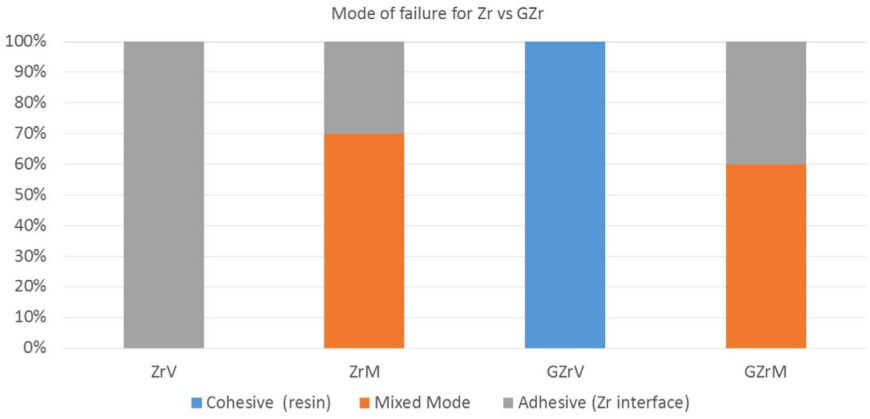

Fig. 4. Showing the modes of failure for glass rods bonded to sandblasted, and glazed + etched zirconia plates respectively, where: "Cohesive" indicates a predominantly cohesive failure in the resin. “Mixed Mode” indicates where cohesive failure occurred in the resin and adhesive failure in the resin-zirconia interface. “Adhesive” refers to the failure that occurred at the zirconia interface.

critical crack length immediately before the initiation of unstable crack within the resin as evident in Fig. 6.

4. Discussion

The purpose of this study was to determine the interfacial fracture toughness between glazed zirconia and two different resin bonding systems, using a fracture energy release approach. The application of a glaze layer followed by hydrofluoric acid etching and silanization with Monobond-S on zirconia, significantly increased ($p < 0.05$) the strain energy release rate of Variolink II resin cement (202.35 J/m^2). The addition of a glaze layer to the zirconia, created a silica-rich surface, which is conducive to the recommended surface treatment protocol for silica-based ceramics [8]. In the unglazed ZrV group, the use of Variolink II resin cement alone on air-abraded zirconia, produced the lowest mean strain energy release rate value amongst the four groups.

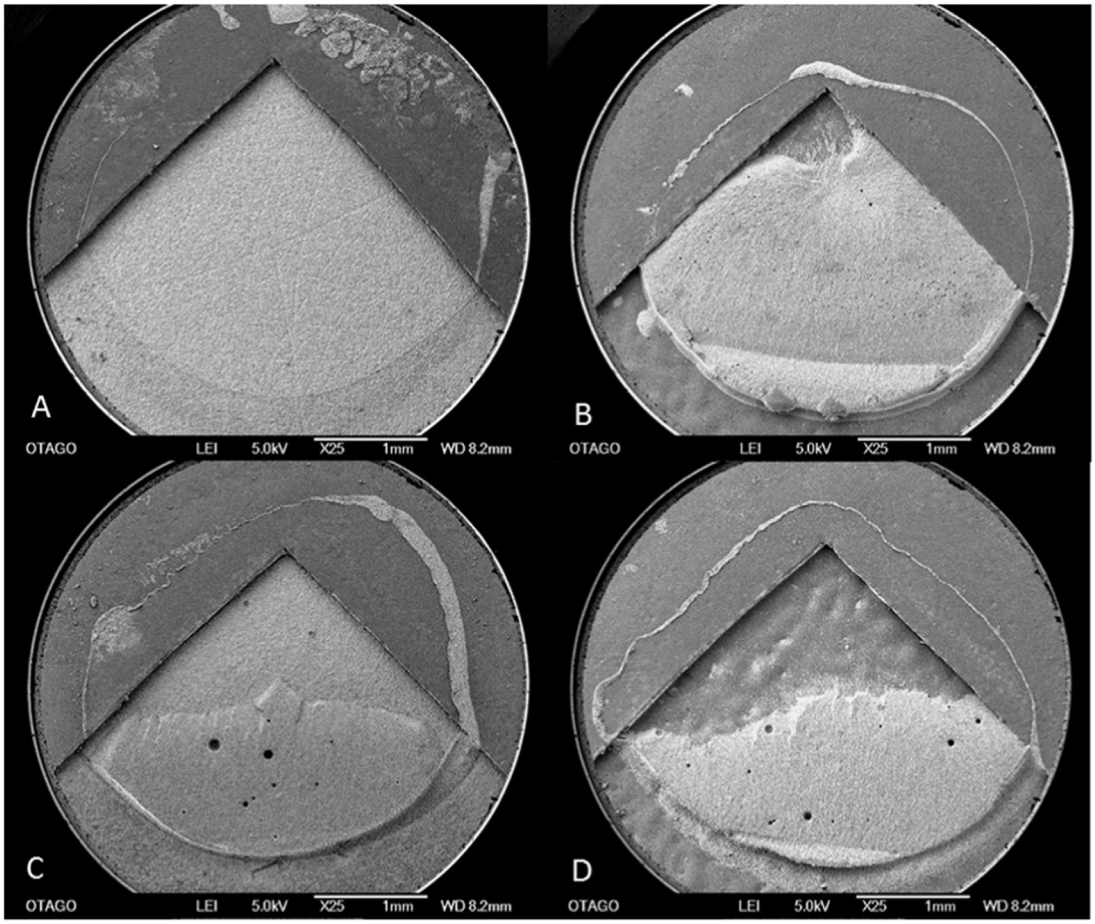

Fig. 5. SEM images of the glass rods after de-bonding from the zirconia adhesive surfaces. Where (A) represents the predominant mode of failure for group **ZrV**, showing adhesive failure at the resin-zirconia interface with smaller crack fronts. (B) Represents the mode of failure of group **GZrV**, showing predominantly cohesive failure at resin interface with a small crack front initiated in the glaze/cement interface, exposing the glazed surface (imprinted) at the chevron tip. (C) Represents the predominant mode of failure (mixed mode) of group **ZrM**, showing adhesive failure at resin interface on the chevron notch with a larger crack front, transferred into cohesive failure within resin in which the fracture resin cement is evident on the glass rod. (D) Represents the predominant mode of failure (mixed mode of group) **GZrM**, showing adhesive failure at resin interface on the chevron notch with a larger crack front similar to group ZrM and surface, transferred into cohesive failure within resin in which the fracture resin cement is evident on the glass rod with an imprint of the glazed zirconia substrate interface.

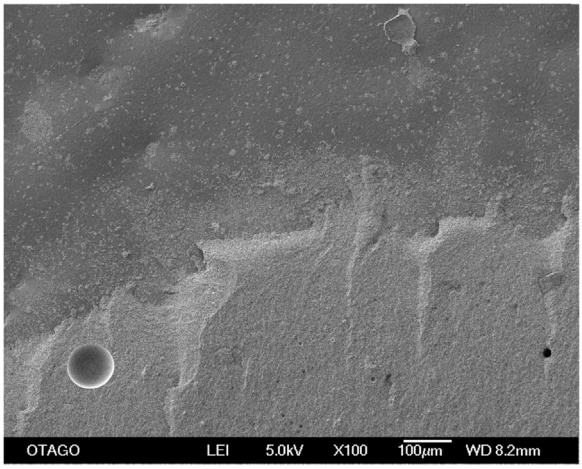

Fig. 6. SEM image, showing the transitional zone observed in group GZrM at 100×, magnification.

This is substantiated by the manufacturer's technical data sheets, which states that Variolink II resin cement lacks a coupling agent that can chemically bond to zirconia. However, the combination of etching with hydrofluoric acid followed by application of a silane coupling agent has been shown to provide the best adhesion to silica-based ceramics [44–46] with good long-term clinical success [47,48]. This is attributed to the hydrolysable functional groups reacting with the surface hydroxyl groups of the silica-based substrate and the organic functional groups, reacting with functional groups of resin cement [27,49]. Due to the chemically inertness of zirconia, it cannot be etched with hydrofluoric acid to create a micromechanically retentive surface for effective bonding [50]. Therefore, the lack of silica renders silane an ineffective coupling agent for oxide-based ceramics and considered an effective wetting agent only [27,28]. However, the deposition of silica in this study, is achieved by glazing with low-fusing glass, which completely covers the surface with a glaze layer. Therefore, the interfacial fracture energy of glazed zirconia is determined predominantly by the glaze and resin bond interface, and not at the zirconia substrate. However, in the unglazed group ZrM, the use of Metal/Zirconia Primer on air abraded zirconia significantly increased the strain energy release rate of Multilink-Automix resin cement (59.40 J/m^2). Multilink-Automix resin cement does not contain phosphate monomer, therefore chemical bond is achieved with Metal/Zirconia Primer containing 6-methacryloxyhexyl phosphonoacetate (6-MHPA). Some studies have shown its coupling effect with zirconia and resin cement to be effective [51,52]. The result from the present study for group ZrM is validated by other studies that show phosphonoacetate acid monomers are more affective for resin bonding to zirconia than we found in group ZrV [35,53,54]. The co-operative effect of phosphate monomer and silane on silica coated zirconia has been known to produce a better bond strength compared to using only silane or MDP alone [55–58]. Tanaka et al. attributed this result to the interaction between the two components that resulted in rapid hydrolysation of the silane coupling agent due to the presence of phosphate monomer. This promotes the formation of a stronger polysiloxane network that is more hydrophobic and resistant to hydrolysis [58]. In the present study, the mean adhesion energy of group GZrM was not significantly different compared to group ZrM and significantly lower than GZrV, suggesting that the combined use of Metal/Zirconia Primer and Monobond-S reduced the silanisation efficacy. Metal/Zirconia Primer contains bisphenol-A ethoxylated dimethacrylate (Bis-EMA), which is a monomer analogous to Bis-GMA containing two aromatic groups but lacking two hydroxyl groups (-OH). Chen et al. reported that the presence of Bis-GMA, a commonly used wetting agent for more hydrophobic cements, inhibits water evaporation from the condensation reaction of silane and decreases the efficacy of the silane coupling effect [59]. Group GZrM received separate applications of Monobond-S followed by Metal/Zirconia Primer. SEM Fig. 5D and in more detail, Fig. 6, shows the fractured surface of group GZrM revealing a transitional zone at the critical crack length before the unstable crack growth, which is also the point where the maximum load is reached. It should also be noted, that similar to urethane dimethacrylates (UDMA) and triethyleneglycol dimethacrylate (TEGDMA), bis-GMA is a commonly used cross-linking monomer in self-etch and etch-and-rinse tooth adhesive systems [60]. However, due to the relatively smooth microstructure of etched and silanised glaze surface compared to dentine, with an exposed collagen fibril network, the formation of a similar hybrid layer is not possible between the resin and ceramic interface. Additionally, since the phosphonoacetate has a limited coupling effect to silica, the unreacted Metal/Zirconia Primer may act as an intermediate unfilled resin layer between the glaze and Multilink-Automix cement, therefore prone to swelling and hydrolysis, which may lead to a further reduction in mechanical properties of the unfilled resin layer to such an extent, that it compromises the adhesion between the ceramic substrate and resin [53,61,62].

Fracture behaviour of an adhesive joint is governed by material properties, distribution of defects, stress and environmental effects, bond efficacy should always be assessed not only from bond strength data alone, but together with a fractographic analysis to better understand and predict bonded interface reliability [63]. The mechanism of fracture toughness and adhesion energy consistently induces a controlled adhesive failure at the crack front and thus the modes of failure manifested will either be mixed interfacial (adhesive failure and cohesive within resin) or failure at adhesive interface [46,64]. In the present study, groups ZrM and GZrM had an inconsistent incidence of adhesive and cohesive failures associated with a predominantly larger crack front. This can be explained by the formation of larger sized air bubble defects from two-paste hand mixing, which can be seen in Fig. 5C and D. On the contrary, group GZrV with the highest strain energy release rate of 202.35 J/m^2 was found to fail consistently within the resin cement, and had a smaller crack front (Fig. 5A). No adhesive failure was observed at the interface between the zirconia substrate and the glaze layer, which further confirms that the weakest link of the glazed specimens was dictated by the fracture toughness and adhesion energy of the resin cement. The glaze was able to consistently resist delamination in its entirety, which indicated the strain energy release rate between zirconia and glaze was higher than between glaze and resin.

5. Conclusion

Within the limitations of this study, the authors found that:

- Glaze coating on zirconia, which has been etched with hydrofluoric acid and the use of silane as a coupling agent, has a significantly higher strain energy release rate in resin adhesion than non-glazed zirconia that was surface treated with phosphate monomer coupling agent.
- Metal/Zirconia Primer enhanced bonding to zirconia surfaces that were not glazed but weakened the bond on etched and silanised glazed surfaces.

Acknowledgments

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