

Full Length Research Paper

General principles for achieving adequate bond to all-ceramic restorations

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Achieving strong and reliable bond to all-ceramic restorations is a pre-requisite for long term clinical success. With the great diversity of the available materials, there is a need for establishing general concepts for bonding all-ceramic restorations. The aim of this study was to evaluate bond strength to two ceramic substrates using different resin cements in combination with different surface treatments. Zirconia and glass ceramic discs received either airborne particle abraded, etched with hydrofluoric acid and coated with silane coupling agent, or combination of particle abrasion and silane coupling agent. Specimens were bonded to composite resin discs and sectioned into micro-bars to evaluate ceramic resin micro-tensile bond strength ($\alpha = 0.05$). Statistical analysis revealed that the type of ceramic substrate (polycrystalline - glass ceramic), type of resin cement (MDP or non MDP containing), type of surface treatment, and their interaction all had a significant influence on ceramic resin micro-tensile bond strength. Combination of adequate micro-mechanical retention (particle abrasion) and chemical bonding (MDP for zirconia and silane for glass ceramic) is a pre-requisite for achieving reliable ceramic resin bond strength. Proper selection of type of resin cement and type of surface treatment that match the ceramic substrate will result in significant improvement of ceramic resin bond strength.

Key words: MTBS, zirconia, glass ceramic, bond strength.

INTRODUCTION

Due to their high esthetic profile, mechanical properties, chemical stability, and biocompatibility; all-ceramic restorations have become the focus of dental practitioners, researchers, and manufacturers alike (Piconi and Maccauro, 1999). In particular, polycrystalline ceramics, such as alumina and zirconia, are becoming widely used as a framework material for construction of all-ceramic fixed restorations (Blatz, 2002). Establishing a good bond to all-ceramic restorations improves their retention, reduces micro-leakages, and enhances the fracture resistance (Blatz, 2004).

Achieving a strong and durable bond to glass ceramics depends on applying hydrofluoric acid (HF) followed by

application of a silane coupling agent (Blatz et al., 2003). Hydrofluoric acid attacks the glass phase producing a retentive surface (Ozcan and Vallittu, 2003) suitable for micromechanical bonding, and the silane coupling agent promotes a chemical bond between the silica phase of these ceramics and the methacrylate groups of the silane coupling agent (Hooshmand et al., 2002; Chaiyabutr et al., 2008). On the other hand, this method is not effective for the glass free zirconium-oxide ceramics as its composition and physical properties makes this material resistant to the acidic or alkaline corrosive materials (Aboushelib et al., 2007).

Silane coupling agents act as adhesion promoters that bond silicone dioxide with the hydroxyl group (OH^-) on the ceramic surface. They are hybrid inorganic-organic bifunctional molecules that also have a degradable functional group that copolymerizes with the organic matrix of

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the resin cement (Aboushelib et al., 2008). Most silane coupling agents also increase the substrate surface energy and improve the surface wettability to resin cements (Debnath et al., 2003). Recent theories about silane coupling agents advocate that they somehow modify the outermost inorganic oxide layer of the substrate promoting better adhesion with resin cements (Child and VO, 1999). However, application of silane coupling agent is not recommended for bonding polycrystalline ceramics as previously mentioned (Ozcan and Vallittu, 2003). Consequently, alternative bonding techniques are required to enhance bonding to zirconia restorations (Aboushelib et al., 2009).

Recent research regarding bonding to zirconia ceramics focused on different approaches. Mechanical retention can be achieved either by adding glass to the surface (Keiichi, 2006), or by modifying the surface structure, for example, with airborne particle abrasion (Kern and Wegner, 1998). On the other hand, chemical bonding to zirconia by using a phosphate ester monomer, 10-methacryloyloxydecyl dihydrogenphosphate (MDP) in combination with airborne particle abrasion showed satisfactory results (Spohr et al., 2008). Airborne particle abrasion with alumina particles and application of a tribochemical silica coating allows for chemical bonding to a silane coupling agent and to resin cement (Piascik et al., 2009), but this procedure is not recommended for glass ceramics as it could lead to micro-cracking and premature failure (Wang et al., 2008). Recently Aboushelib et al reported high bond strength values utilizing selective infiltration etching and a novel silane-based zirconia primer (Aboushelib et al., 2009). Very recent studies showed that selection of suitable cement for luting zirconia was the most important factor when compared to other variables such as the surface treatments (Oyague et al., 2009; Oyague et al., 2009; Nothdurft et al., 2009).

Currently, there are insufficient data about the actual mechanism of reaction of the MDP monomer and whether it establishes a true chemical bond with zirconia or whether it relies basically on the micro-retention provided by particle abrasion. Furthermore, information is lacking on bond strengths of resin composite luting cements to non-etchable ceramics, and in particular, there is scarce in the available silane comparison reports (Amaral et al., 2008). The aim of this study was to evaluate micro-tensile bond strength of two types of resin cements to different ceramic substrates using different surface treatments.

MATERIALS AND METHODS

This study evaluated the micro-tensile bond strength between two types of ceramics (zirconia and glass ceramic) and two types of resin cements: MDP containing (Panavia 21, Kuraray, Japan) and non-MDP containing (Rely X ARC, 3M-ESPE, Seefeld, Germany) using three surface treatments (airborne particle abrasion, application of hydrofluoric acid with a silane coupling agent, and combination of airborne particle abrasion with HF acid and silane

application). Materials properties are summarized in Table 1.

Preparation of the specimens

Thirty cylinder-shaped (\varnothing 19.5 x 3 mm high) Zirconia sintered discs (Procera Zirconia, NobelBiocare, Goteborg, Sweden) and 30 cylinder-shaped (\varnothing 19.5 x 3 mm high) CAD/CAM leucite-filled porcelain (ProCad, Ivoclar-Vivadent, Schaan, Liechtenstein) were prepared according to the manufacturer's instructions. The bonding surface of each disc was polished using silicon carbide paper (grit 300, 400 and 600) on a rotating metallographic polishing device (Isomet 1000, Buehler Ltd, Lake Bluff, IL,) under water-cooling. The prepared surface was ultrasonically cleaned in 90% ethyl alcohol for 30 min.

Sixty composite resin cylinders discs (\varnothing 19.5 x 3 mm high) were prepared by incrementally filling a plastic mold with a microhybrid resin composite (Tetric Evo Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein). The discs were prepared in a plastic mould (3 mm in depth and 19.5 mm in diameter). Each composite layer was light polymerized for 40 s with an LED light-curing unit (Elipar Freelight 2, 3M-ESPE, Seefeld, Germany) with a minimum light intensity of 700 mW/cm² which was frequently checked and the unit was re-charged as required. The bonding surface of the composite resin discs received the same previously mentioned surface treatment followed by ultrasonic cleaning in distilled water for 30 min.

Surface treatment

The prepared ceramic discs were divided into three groups according to the following surface treatment (Table 2):

- Airborne particle abrasion with 50 μ m aluminum oxide particles at a pressure of 1.5 bars and at a distance of 2 cm followed by ultrasonic cleaning in distilled water for 30 min.
- Application of HF acid and Silane coupling agent following manufacturer's instructions for each resin cement (Table 1).
- Airborne particle abrasion in combination with application of HF acid and Silane coupling agent following the manufacturer's instructions.

Bonding of the specimens

Luting procedures were carried out by mixing each resin cement according to the manufacturer's instructions then coating it on the surface of the ceramic discs followed by seating of the composite discs under a fixed load of 300 g using a loading jig. Excess cement was wiped off and the specimens were light polymerized for 40 s from the top and bottom surfaces. Specimens that received a silane coupling agent were allowed to dry for 10 min in open air before application of resin cement. Bonded specimens were stored in a laboratory oven at 37°C and 100% relative humidity for 4 weeks.

Micro-tensile bond strength test

After 2 months, all bonded specimens of each group were vertically sectioned under running water into micro-bars (1 mm thick and 6 mm long) using a precision cutting machine (Isomet 1000; Buehler, Lake Bluff, Ill); 25 sound micro-bars were obtained for each test group (n = 25). The sectioned micro-bars were stored in distilled water in an oven at 37°C and 100% relative humidity. After 30 days those microbars were retrieved and micro-tensile bond strength was performed: each micro-bar was bonded to the attachment unit grip

Table 1. Composition and manufacturer of the used materials.

| Material | Composition | Manufacturer |
|-----------------------------------|---|---|
| Procera Zirconia | ZiOx 77% / Ytria 20% / Hafnium 2% / Silica <1% | Nobel Biocare, Goteborg, Sweden |
| ProCad | leucite-filled porcelain, inorganic pigments | Ivoclar Vivadent, Schaan, Liechtenstein |
| Panavia 21 TC | BPEDMA/MDP/DMA/ silanized barium glass and silica particlesr, benzoylperoxide, TPBSS, N,N-diethanol p-toluidine | Kuraray, Osaka, Japan |
| Clearfil Porcelain Bond Activator | Dimethacrylaat monomeer, silane-coupler | Kuraray, Osaka, Japan |
| Clearfil SE Bond | MDP, HEMA, Hydrophilic dimethacrylate, dl-Camphorquinone, N,N-Diethanol-p-toluidine, water | Kuraray, Osaka, Japan |
| RelyX ARC | BisGMA, TEGDMA, zirconia/silica fillers | 3M, Minnesota, Verenigde Staten |
| 3M ESPE Ceramic Primer | Pre-hydrolyzed silane-coupling agent, alcohol and water | 3M ESPE, Minn, USA |
| Tetric EvoCeram | UDMA, Bis-GMA, TEGDMA | Ivoclar Vivadent, Schaan, Liechtenstein |
| Ultradent Porcelain Etch | Hydrofluoric acid 9.5% gel | Ultradent Products, Inc. USA |

Table 2. Ceramic resin micro-tensile bond strength (MPa) using different surface treatments.

| Cement | Ceramic | Surface | MTBS (MPa) | Std. Deviation | Failure Mode |
|------------|----------|----------------------------|-------------------|----------------|---------------------------------------|
| Panavia 21 | zirconia | Particle abrasion + silane | 31.5 | 3.5 | Cohesive in resin cement. |
| | | Particle abrasion | 28.8 | 2.0 | Interfacial a cross bonded interface. |
| | | Silane | 26 | 2.2 | Interfacial a cross bonded interface. |
| | Procad | Particle abrasion + silane | 19.9 | 1.6 | Cohesive in resin cement. |
| | | Particle abrasion | 8.9 ^A | 2.3 | Interfacial a cross bonded interface. |
| | | Silane | 20.6 | 2.1 | Interfacial a cross bonded interface. |
| RelyX ARC | zirconia | Particle abrasion + silane | 13.7 | 2.1 | Interfacial a cross bonded interface. |
| | | Particle abrasion | - | - | Interfacial a cross bonded interface. |
| | | Silane | - | - | Interfacial a cross bonded interface. |
| | Procad | Particle abrasion + silane | 16.7 ^A | 2.2 | Interfacial a cross bonded interface. |
| | | particle abrasion | 23.7 | 2.4 | Interfacial a cross bonded interface. |
| | | Silane | 5.8 ^{Ba} | 1 | Interfacial a cross bonded interface. |

There was a significant difference ($P < 0.001$) between Panavia 21 and RelyX ARC for both types of ceramics and for all surface treatments. Groups with superscript letter indicate significant difference in MTBS values between different surface treatments for one type of ceramic using one resin cement.

using an adhesive resin (Clearfil SE Bond, Kuraray, Japan) and stressed to failure in tension using a universal testing machine (Instron 6022, Instron Limited, High Wycombe, UK) at a crosshead speed of 0.5 mm/min. After fracture, the specimens were ultrasonically cleaned and examined under scanning electron microscopy (SEM) using different magnifications (XL30, Philips, Eindhoven, the Netherlands). Data were examined and analyzed using one and two-way analysis of variance test (ANOVA) using computer software (SPSS 10, SPSS Inc, Chicago, USA). Bonferroni *post hoc* test was used for pair-wise comparisons. Based on the number of specimens ($n = 25$), small effect size ($F = 0.25$), and 0.05 level of significance, the test of choice had adequate power (1.0) to detect significant differences in micro-tensile bond strength between the tested groups (Oyague et al., 2009).

RESULTS

Analysis of the data revealed that there were significant differences in the micro-tensile bond strength values which were influenced by the type ceramic substrate ($F = 229$, $P < 0.001$), type of resin cement used ($F = 379$, $P < 0.001$), type of surface treatment ($F = 50$, $P < 0.001$), and their interactions ($F = 30$, $P < 0.001$) (Table 2). Micro-tensile bond strength values observed using Panavia 21 were significantly higher compared to RelyX ARC for both ceramic substrates and for all types of surface treatment (except combination of proCad discs, Panavia and particle

abrasion).

For zirconia, the combination of particle abrasion and silane coupling agent resulted in significantly higher bond strength values compared to the other surface treatments. Moreover, the combination of particle abrasion and silane coupling agent was the only method where specimens bonded using RelyX ARC survived the sectioning procedure. For ProCad discs, combination of Panavia 21 and silane coupling agent produced higher bond strength value, meanwhile using RelyX ARC and airborne particle abrasion produced the highest bond strength values.

SEM observation of the fractured zirconia specimens revealed that the combination of airborne particle abrasion and silane coupling agent resulted in cohesive failure pattern meanwhile all other test groups demonstrated an interfacial failure pattern leading to exposure of the zirconia surface at the bonded interface (Figures 1A and 1B). For ProCad discs, an interfacial fracture pattern was observed for all specimens bonded with both resin cements. Areas demonstrating remnant of resin cement where some times observed on the fractured surfaces (Figure 2).

DISCUSSION

Microtensile test allows for appropriate alignment of the specimens, homogeneous distribution of stress, and testing of the bonded interface in direct tension which allows for sensitive comparison or evaluation of bond strength values (Aboushelib et al., 2007). Alternatively, shear tests have been criticized for the development of non-homogeneous stress distributions in the bonding interface, inducing either under-estimation or a misinterpretation of the results, since failure often starts in one of the substrates and not at the adhesive zone (DeHoff et al., 1995).

The findings of this study clearly illustrates that the bond strength between ceramic substrates and resin cements is sensitive to several variables as the type of adhesive used, the method of surface treatment and their interaction. Panavia 21 demonstrated in general higher bond strength values compared to Rely X ARC. Such fact could be directly related to differences in the chemical composition (namely MDP), difference in filler content which could affect the wetting ability of the cement, and to differences in their mechanical properties as resin cement with weaker cohesive strength is expected to fail at lower loads, Table 1.

For zirconia ceramics, it was observed that the combination of particle abrasion and silane coupling agent produced the highest bond strength values and was associated with cohesive type of failure which was not observed for other test groups. Such fact was previously observed for tribochemically coated zirconia as the silane coupling agent was able to bond to the silica content of the

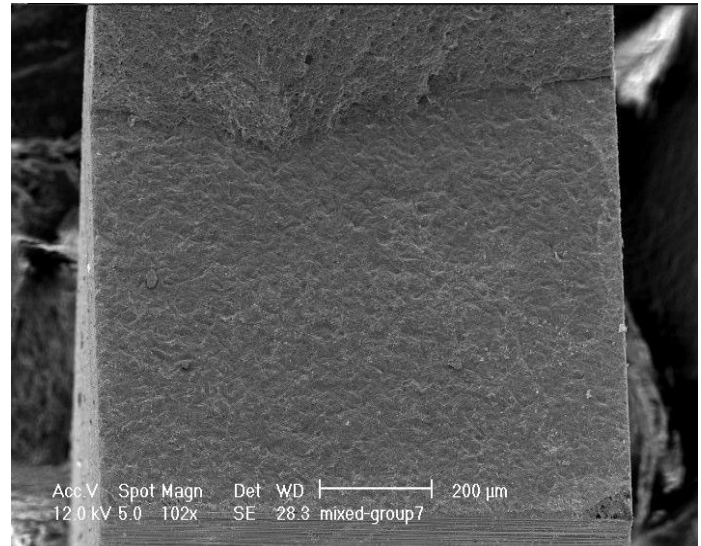


Figure 1-A. SEM image, 100x, demonstrating interfacial fracture of airborne particle abraded zirconia specimen bonded with Panavia 21. Observe the exposed zirconia surface.

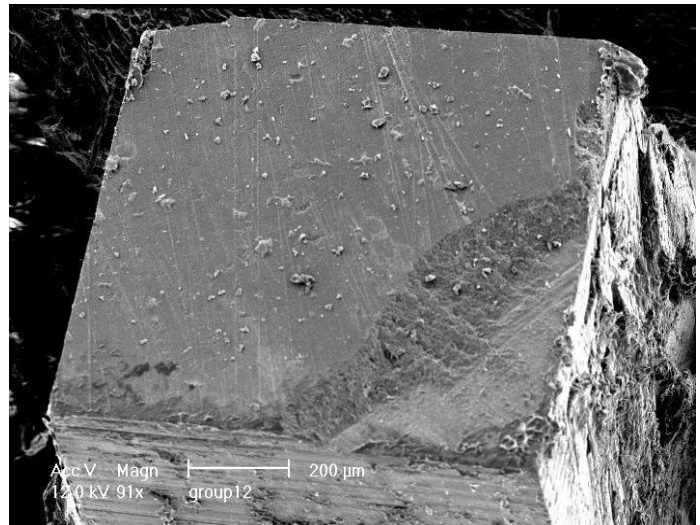


Figure 1-B. SEM image, 100x, demonstrating cohesive fracture of particle abraded and silanized zirconia specimen bonded with Panavia 21. Fracture occurred in resin cement.

coating (Matinlinna et al., 2006; Matinlinna et al., 2007; Blatz et al., 2007) but was not recommended for directly bonding to zirconia as spontaneous failure of the specimens was observed with water storage (Wegner and Kern, 2000). Such contradiction could be explained on the basis that the applied silane coupling agent was able to enhance wetting of the MDP containing resin cement resulting not only in higher bond strength values but also in cohesive failure as well. The phosphate ester group of the MDP is reported to directly bond to metal oxides (Luthy et al., 2006), forming a strong poly-molecular film

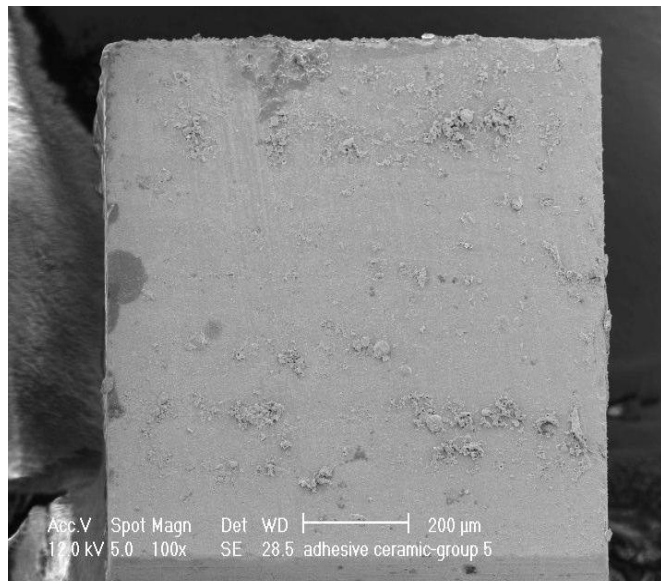


Figure 2. SEM image, 100x, demonstrating interfacial failure in ProCad discs which was observed for all specimens bonded with both cements using either of the surface treatments.

that react with the substrate by the acidic monomer. The mix of resin bonding and silane coupling agent (Clearfil SE bond mixed with Clearfil Porcelain Activator) with phosphate monomer MDP may have promoted the bonding mechanism by improving surface wettability and forming cross-linkages with methacrylate groups as well as siloxane bonds with the OH-groups of the ceramic surface which is in concordance with previous studies (Oyague et al., 2009; Blatz et al., 2004). On the contrary, bonding zirconia with a non MDP conventional Bis-GMA cement (Rely X ARC) recorded lower bond strengths values than the MDP containing resin cement (Panavia 21). As in the other situations Rely X ARC recorded 100% of premature debonding after water storage, which was in agreement with other studies (Kern and Wegner, 1998; Luthy et al., 2006). The absence of adhesive functional monomers in the cement composition may explain the lack of chemical bonding (Kern and Wegner, 1998).

The results of this study showed that the principal type of failure was interfacial (adhesive) for the ProCad discs independent of the resin-cement and surface treatment, this could be due to hydrolysis of the bonding between the silica content of the ceramic and the hydroxyl terminal of the silane (Oyague et al., 2009). On the other hand the cohesive failure observed in case of particle-abraded zirconia may be due to the silane enhanced wetting of the adhesive resin and thus no bond hydrolysis was expected for this interface. On the contrary, MDP monomer is known for its chemical stability due to the presence of a long carbonyl chain (Oyague et al., 2009; Valandro et al., 2007).

The high value bond strength of Panavia to ProCad is a result of chemical and micro-mechanical bonding while significant drop was noted when silane was not applied which is in accordance with previous studies (Chaiyabutr et al., 2008; Kumbuloglu and Lassila, 2005; Bailey, 1989). In contrary when the Bis-GMA resin cement Rely X ARC was used the lowest values were observed in the presence of silane, this may be due to water sorption and lost of the bond between the silane and the resin (Karabela and Sideridou, 2008).

Conclusion

Within the limitations of this *in vitro* study, the following conclusions could be drawn:

The phosphate monomer- containing luting cement is recommended for bonding to zirconia restorations. In combination with particle abrasion and silane application, it will reduce chance of interfacial fracture (debonding failure).

Bonding to glass ceramic relies on both micro-mechanical (HF etching) and chemical bonding (silane). Silane coupling agent improved wetting and bonding of resin cements.

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