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Adhesion between zirconia and indirect composite resin

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A R T I C L E I N F O

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A B S T R A C T

Purpose: The purpose of this study was to determine the bond energy using the strain energy release rate of three indirect restorative composite veneering systems to YZr.
Materials and methods: Three indirect composite veneering systems (Ceramage – Shofu Inc.; Signum – Heraeus Kulzer GmbH; Sinfony- 3M ESPE) were bonded to YZr stabilized zirconia plates with and without sandblasting and manufacturer's recommended bonding agents per the method described by Cheng et al. [32] consisting of two opaque layers on the YZr plate at the bond surface interface and a 12 mm composite rod. The specimens were brought to failure with a universal testing machine and *G*-values calculated. One-way ANOVA and Dunnetts's test ($P=95\%$) were performed. Homogeneity of the variables was confirmed with Bartlett's test.
Results: No significant difference was observed between the *G*-values for the control groups of Ceramage, Signum and Sinfony. Within the Ceramage group, there was no significant difference between the surface treatments. The Signum group showed a significant difference between the control and sandblasted groups as well as the sandblasted surfaces in combination with bonding agent groups, but no significant difference between control and bonding agent alone. The Sinfony group, showed no significant difference between the control and sandblasted groups, but a significant difference between the control and sandblasted with bonding agent groups (Rocatec).
Conclusion: The application of acidic functional phosphate monomer MDP or silicatising the YZr surfaces before veneering with indirect composite veneering material produced higher bond energy. Sandblasting the YZr surfaces with 120 grit AlO_2 only, did not increase the bond energy.

1. Introduction

Yitrium stabilized Zirconia Ceramic (YZr) has been developed as a framework material for tooth-supported or implant-supported all-ceramic restorations and implant abutments. This is due to its biocompatibility, low bacterial adhesion, high strength and natural esthetic properties [1–5].

Failures, with and without exposing the underlying framework, in the form of chipping within the veneering porcelain have been reported in the literature [5–12]. As an alternative to porcelain, Komine et al. reported on the use of composite resin as a viable veneer system. An additional advantage of using composite resin was the energy absorption of composite resulting in a preferable tactile response in natural teeth opposing an implant [1,12,13].

However, due to the chemical inertness of YZr, bonding remains problematic [11,14,16–23]. Limited research is available on the bond strength between proprietary indirect composite systems and YZr; published results from which were predominantly obtained by using the shear bond test [1,14]. Yet, data from these measurements, using the concept of nominal stress, are inconsistent and contain large deviation in test results between laboratories [15]. In fact, the mechanics of the nominal shear bond test, draws more criticism than approval. In a study done by Della Bona and van Noort finite element analysis (FEA) was used to demonstrate that tensile and shear bond strength measurements were highly dependent on the geometry of the test arrangement; the nature of the load applied; film thickness of the adhesive; as well as the elastic modulus of the materials involved [15].

By using the fracture energy release rate approach, it is possible to determine the potential power of stable crack propagation within an interface. This can be achieved using G_{Ic} (strain energy release rate) which is the amount of energy required to separate two bonded materials [25]. This approach takes into account, the mechanical properties of the adhesive material and the geometries of the test arrangement and adhesive surface.

Therefore, the purpose of this study was to determine the bond energy using the strain energy release rate, of three proprietary indirect restorative composite veneering systems to YZr.

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2. Materials and method

Three proprietary indirect composite veneering systems, incorporating their dentine and opaque, were bonded to zirconia plates using their manufacturer's recommended bonding agents (Table 1) and preparation techniques (Table 2). 132 YZr rectangular plates were sectioned from milling blocks, using a diamond grit blade on a low speed cutting machine (DTQ-5, Laizhou Huayin Testing Instrument Co., Ltd., Shangdong, China) under water irrigation. Prior to sintering the plates were hand polished using 400 grit Silicon Carbide abrasive paper (Struers, Denmark) to ensure a flat veneering surface.

All the plates were veneered according to each individual manufacturer's instructions with a composite rod as described by Cheng et al. [32] consisting of two opaque layers at the bond surface interface and a 12 mm composite rod. The geometry of the chevron shaped bond interface (Fig. 1) was adapted from Tantbirojn et al. [33]. The chevron shaped bonding surface was created by applying a custom-made cutout sticker, peeled off a preprinted sheet of ~50 micron non-stick polymeric transparent PVC film (Grafiprint; Houthalen, Belgium) produced by a commercial printing company, ensuring that each chevron notch shape was exactly the same for each specimen. A precision glass tube, inside diameter of 4 mm, lined with a thin film of petroleum jelly to prevent adhesion, was positioned over the chevron-shaped bonding area and incrementally filled with indirect composite resin. After polymerization, the glass tube was removed.

The specimens were loaded (Fig. 2) 10 mm from the bonded interface at a cross-head speed of 0.5 mm/min in a universal testing machine (Instron, model 3369, Instron Corp. Canton, MA, USA). The load at failure (F_{max}) was recorded using a 1 kN load cell and Istron Bluehill 3 software (Instron Corp. Canton, MA, USA).

Strain energy release rates were calculated using the formula by Cheng et al. [32]

$$G_{Ic}(\mathrm{J/m^2}) = 104.5(F_{max})^2 L^3 / ED^6$$

where F_{max}=Load at failure (N), L=Distance to loading point (mm), E=Elastic modulous of the composite cylinder (dentine

Table 2
YZr specimen preparation process (n=12 per test group) prior to veneering with indirect composite resin.

Material	Sandblasted with 120 μm grit ALO_2, 2 bar pressure.	Bonding agent
Ceramage		
CER1 (Control)	No	No
CER2	No	AZ primer
CER3	Yes	No
CER4	Yes	AZ primer
Signum		
SIG1 (Control)	No	No
SIG2	No	Zirconia bond 1&2
SIG3	Yes	No
SIG4	Yes	Zirconia bond 1&2
Sinfony		
SIN1 (Control)	No	No
SIN2[a]	N/A	N/A
SIN3	Yes	No
SIN4	Yes	Sandblasted again with Rocatec and ESPESil applied

[a] Sinfony incorporates the use of Rocatec (3M ESPE, USA) sandblasting as part of the bonding system. This process is reflected in group SIN4 and therefore no specimens where prepared for this surface treatment option as per CER_2 and SIG_2.

Table 1
List of proprietary indirect composite veneering systems (dentine, opaque and bonding agent) by material type/trade name (including dentine Elastic modulus), lot number, constituents, and reference for constituents and name of manufacturer.

Material/trade name	Lot	Constituents	Reference for constituents	Manufacturer
Zirconia ceramic				
Vita In-Ceram YZ for in lab (E-modulus 210 GPa)	35760	91% Zirconium oxide (ZrO_2), 5% yttrium oxide (Y_2O_3), 3% hafnium oxide (HfO_2), small amounts (1%) of aluminum oxide (Al_2O_3) and silicon oxide (SiO_2)	Bottino et al. [26]	VITA Zahnfabrik, Germany
Indirect composite materials				
Ceramage dentine (E-modulus 10.7 GPa)	041024	UDMA (Urethane dimethacrylate)	Soancă et al. [27]	Shofu Inc., Kyoto, Japan
Ceramage opaque	100906	UDMA, aluminum silicate, 2-HEMA, glass, pigment, others	Muratomi et al. [28]	Shofu Inc, Kyoto, Japan
AZ Primer (Ceramage)	071213	6-MHPA(6-methacryloxyethyl phosphonoacetate.), acetone, others	Ural et al. [16]	Shofu Inc, Kyoto, Japan
Signum dentine (E-modulus 3.5 GPa)	01300	Bis-GMA(2,2′-bis-[4-(methacryloxypropoxy)-phenyl]-propane) and TEGDMA (Tri (ethylene glycol) dimethacrylate)–SiO_2, Ba–Al–Si (1,0 μm) -	Janda et al. [29] Alves et al. [30]	Heraeus Kulzer GmbH, Hanau, Germany
Signum Opaque F	010209	Multifunctional dimethacrylates; Pyrogenic SiO_2 Photoinitiator Camphorquinone; TiO_2, iron oxides	Janda et al. [29]	Heraeus Kulzer GmbH, Hanau, Germany
Zirconia bond I+II	010021	Bond I: Acetone, 10-MDP (10-methacryloyloxy-decyl-dihydrogenphosphate), acetic acid 010106 Bond II: Methyl methacrylate, diphenyl(2,4,6- trimethylbenzoyl) phosphanoxide	Ural et al. [16]	Heraeus Kulzer GmbH, Hanau, Germany
Sinfony dentine (E-modulus 3.1 GPa)	449469	Microhybrid composite containing: strontium aluminum borosilicate glass, pyrogenic silica, glass ionomer, a mixture of aliphatic and cycloaliphatic monomers	Alves et al. [30] Özcan et al. [31]	3M ESPE, Minnesota, USA
Sinfony opaque powder	445066	3-(trimethoxysilyl)propyl, titaniumdioxide, calcium fluoride, dilauroylperoxide, 1,1,1-trimethyl-N-(trimethylsilyl), silaneamine, hydrolyzation products with silica, iron oxide	Özcan et al. [31]	3M ESPE, Minnesota, USA
Sinfony opaque liquid	518929	bis(methylene)diacrylate, MMA, vinylchloride–vinylacetate copolymer, trimethylbenzoyl-diphenylposphone oxide	Özcan et al. [31]	3M ESPE, Minnesota, USA
Rocatec	–	Silicatized aluminum oxide particles	Bottino et al. [26] Özcan et al. [31]	3M ESPE, Minnesota, USA
ESPE Sil	495219	3-methacryloxypropyltrimethoxysilane(MPS) in ethanol	Özcan et al. [21]	3M ESPE, Minnesota, USA

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composite material), and D=Diameter of the composite cylinder (4 mm).

Statistical analysis was done using STATA software (StatCorp LP, Texas, USA). The data was log transformed for better distribution. One-way ANOVA (P=95%) was performed to determine significant differences between the G-values of the test groups for each material type. Homogeneity of the variables was confirmed with Bartlett's test. Dunnett's test was performed to determine statistical difference (P=95%) to the control within the material groups.

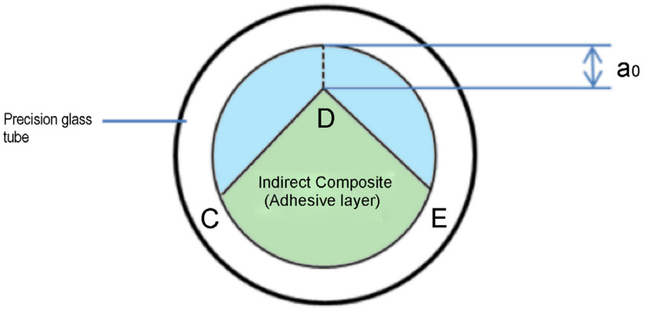

Fig. 1. Geometry of the chevron shaped bond interface (green) as adapted from Tantbirojn et al. [33]. The blue represents the un-bonded surface area created by the non-stick polymeric transparent PVC film. Diameter 4 mm; CDE angle=90°; a_0=0.6 mm. (For interpretation of the references to color in this figure, the reader is referred to the web version of this article.)

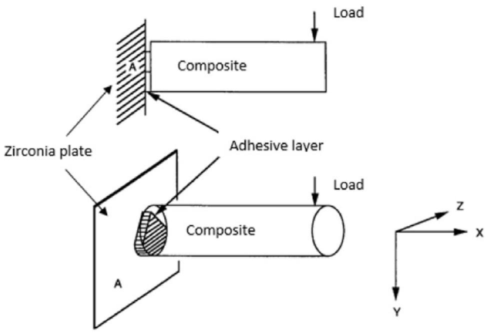

Fig. 2. Schematic diagram of the bond strength measurement test method (adapted from Cheng et al. [32]).

After de-bonding, the percentage surface area mode of failure (adhesive, cohesive and mixed mode) was established using a stereoscopic zoom microscope (SMZ800, Nikon Corporation, Tokyo, Japan). Selected specimens, highest and lowest G values, were qualitatively analyzed under scanning electron microscope (SEM)(JSM 6700 FESEM, JEOL, Japan) to confirm the mode of failure identified with the light microscope and illustrate the differences in surface treatments prior to bonding.

3. Results

3.1. Bond strength test

The mean bond adhesive energy, $G_{Ic,}$ values are shown in Fig. 3. The statistical comparison between groups showed: There was no significant difference between the G values for the control groups of Ceramage, Signum and Sinfony; within the Ceramage groups, there was no significant difference between the surface treatments; within the Signum groups, there was a significant difference between the control and sandblasted and sandblasted with bonding agent, but no significant difference between control and bonding agent; within the Sinfony group, there was no significant difference between the control and sandblasted, but a significant difference between the control and sandblasted with bonding agent (Rocatec).

3.2. Microscopy analysis

The modes of failure are shown in Fig. 4. Adhesive failure occurred when the failure occurred at the interface between the composite system and the zirconia surface. Cohesive failure occurred within the composite system. The high strength properties of the zirconia prevented any cohesive failure within itself. Mixed mode failure was a combination of adhesive and cohesive.

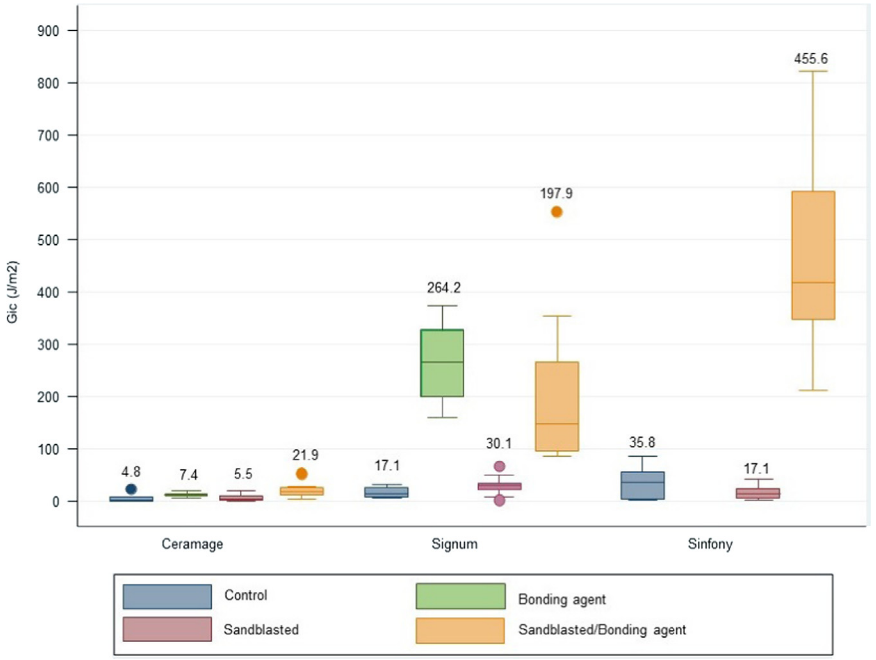

Fig. 3. Mean bond strength (strain energy release rate – G_{Ic}) according to material type, sandblasting, bonding agent and combination of sandblasting with bonding agent. Mean G-values (J/m^2) are recorded above each bar.

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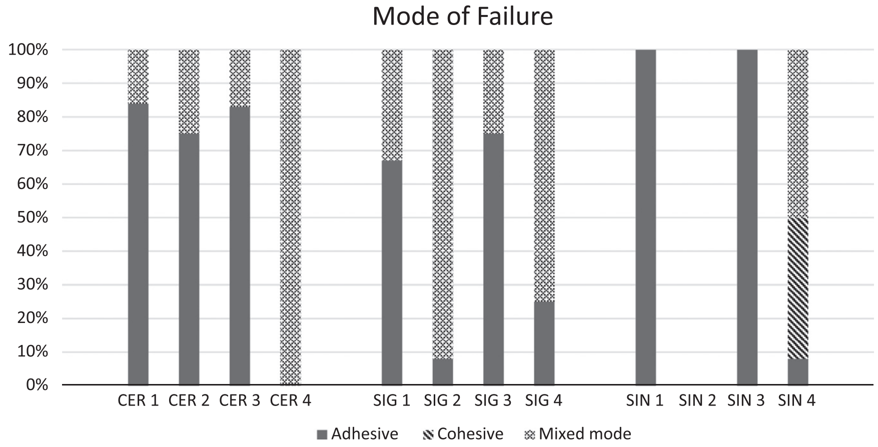

Fig. 4. Mode of failure between the indirect composite and Zirconia plates.

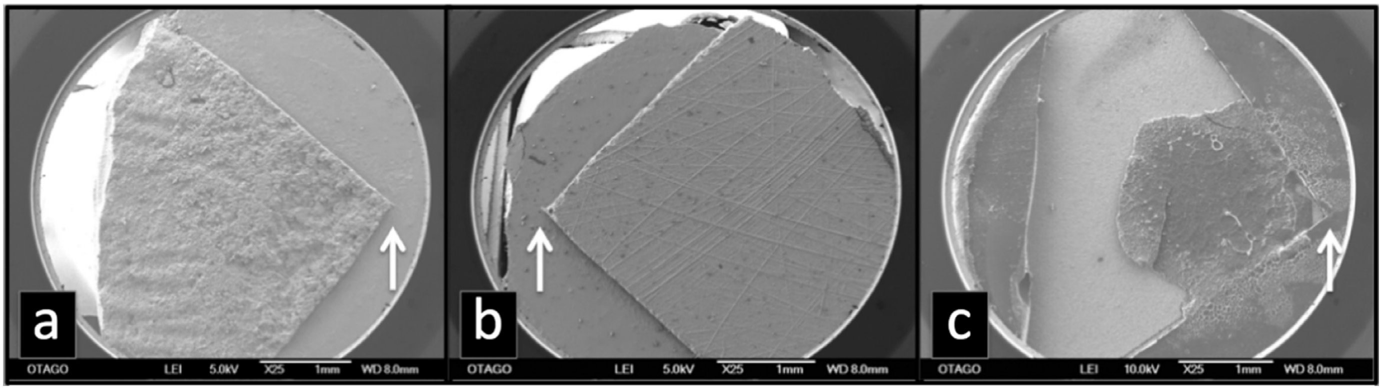

Fig. 5. Fig. 4. Shows low magnification SEM images of typical mode of failure for the various treatment options. The arrows indicate the tip of the chevron notch from where crack propagation initiated. (a) Cohesive failure within the composite material from the specimen group SIN4 (composite rod). (b) Adhesive failure between the composite and zirconia plate taken from specimen group SIN1 (composite rod). (c) Mixed-mode failure between the composite and zirconia plate taken from specimen group CER4 (YZr plate).

3.3. *SEM analysis*

SEM analysis confirmed the mode of failure results obtained from the light microscope.

(Fig. 5). SEM images of the YZr surface treatment options prior to composite resin application are shown in Fig. 6.

4. Discussion

In this study, the bond energy release rate was measured between indirect veneering composite systems and YZr, with and without sandblasting and the application of different bonding agents. The results show that the bond energy release rate were influenced by the application of different bonding agents and surface treatments. In addition, the mode of failure was established whereby an adhesive failure would indicate that the interfacial bond within the composite system being tested was weaker than the intrinsic strength of the composite material and a cohesive failure would indicate that the interfacial bond was stronger than the intrinsic strength of the composite material. A mixed mode would show a combination of both.

A relationship was observed between the *G*-value and the types of bonding agent. The low *G*-values recorded in all the control groups, where the composites were bonded to the YZr plates without sandblasting or bonding agents, was expected based on the reports by Komine et al. [14]. This was also confirmed by the predominantly adhesive mode of failure indicating that without the use of a bonding agent and/or sandblasting to increase the surface area, there was no chemical and minimal mechanical bond between the composite systems and the YZr surface (Fig. 4).

A mixed result was recorded in the second treatment option where only the bonding agent was applied to the YZr surfaces. CER2 recorded a low mean *G*-value (7.4 G_{Ic}/Jm^2), whereas SIG2 recorded a significantly higher mean *G*-value (264.2 G_{Ic}/Jm^2). According to previous reports the application of acidic functional monomer containing carboxylic anhydride (4-META) in combination with phosphoric acid (6-MHPA) [12,34,35], or phosphate monomer (MDP) and MDP and silane [36] can yield a durable bond strength between indirect veneering composite and YZr. This corresponded with the SIG2 results where Zirconia Bond I+II, which is a functional Phosphate monomer (MDP), was used. This indicates that the application of an MDP primer (10-methacryloyloxy-decyl-dihydrogenphosphate) containing resin improved the bond between the indirect composite resin and YZr. Özcan et al., explains this as a reaction between hydroxyl groups in the MDP and the zirconia ceramic, as the result of bonding of a phosphate ester monomer to metal oxides such as chromium, nickel, aluminum, and zirconium dioxides [21]. A possible explanation of the low CER2 results is the absence of the carboxylic acid component (4-META) in combination with an acidic functional monomer containing phosphoric acid (6-MHPA) in the AZ Primer (Table 1). Further evidence of the above was the predominantly adhesive mode of failure of the CER2 group whereas the SIG2 group produced a predominantly mixed mode of failure (Fig. 4).

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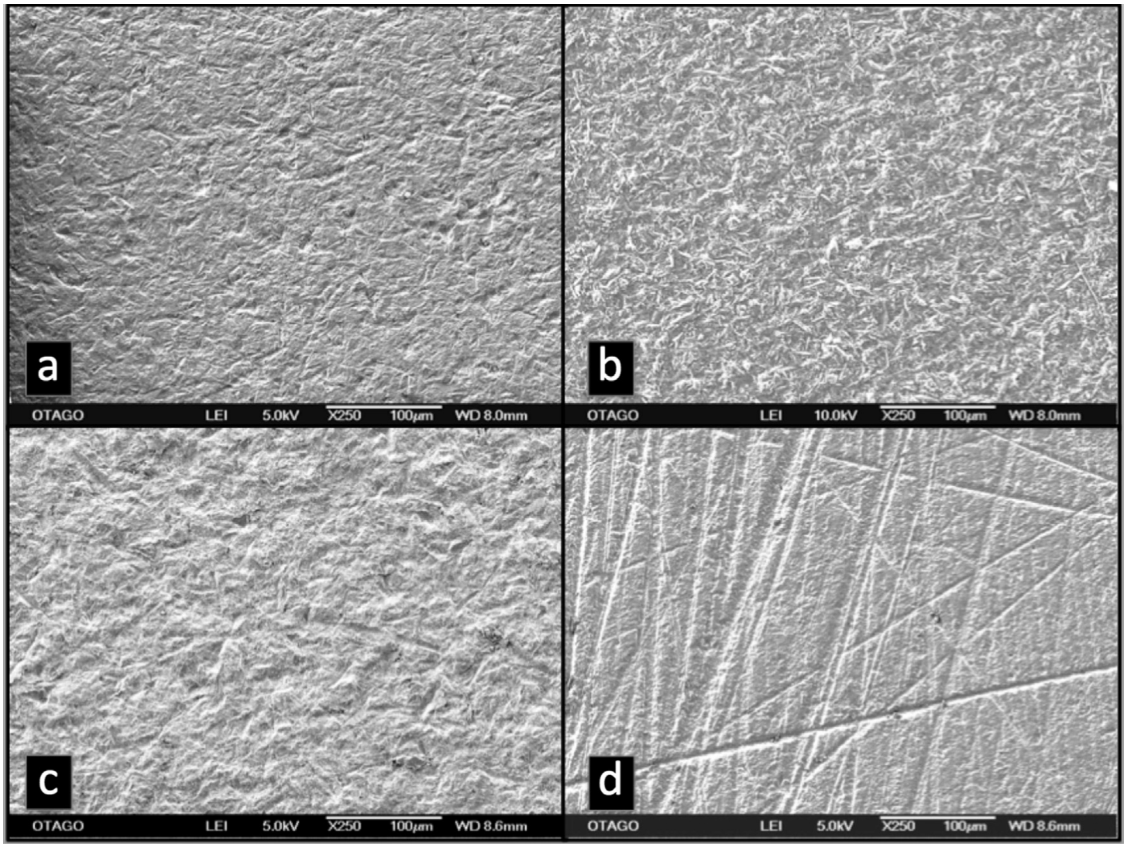

Fig. 6. Shows SEM images (250x) of the YZr surface treatment options prior to composite resin application. (a) sandblasted YZr treated with Zirconia Bond 1&2, (b) sandblasted YZr treated with AZ primer, (c) sandblasted YZr treated with Rocatec and ESPE Sil, and (d) Sintered YZr that was hand polished with 400 grit silicon carbide abrasive paper prior to being sintered.

Sandblasting is a popular method to increase the surface area and roughness for improved interfacial bond. However, sandblasting the YZr bonding surfaces prior to veneering (CER3, SIG3 and SIN3) with composite resin and without the use of a bonding agent, showed no significant increase in the *G*-values compared to the controls despite the larger surface area available for bonding. This is in contradiction to a study by Komine et al. who reported that sandblasting at 0.1 MPa pressure or higher, yields satisfactory initial and durable bond strengths between an indirect composite material and zirconia ceramics [14]. The reason for this difference may be explained by the test method used by Komine and coworkers, which was a shear bond approach. By sandblasting, the surface morphology will change from smooth to roughened and thereby present more surfaces perpendicular to the direction of shear loading and therefore more shear resistance. In contrast, the opening mode of the fracture energy release rate approach does not encounter perpendicular resistance, only increased area available for adhesive bonding. The mode of failure also reflected this finding in that the CER1 and CER2 groups plus SIN1 and SIN2 groups showed the same mode of failure respectively while the SIG3 group mode of failure was similar to the SIG1 group with a slightly higher proportion of adhesive failure mode (Fig. 4).

In the fourth treatment option (CER4, SIG4 and SIN4) where the specimens were sandblasted prior to application of the bonding agent, SIG4 and SIN4 showed a significant increase in bond energy compared to their controls and SIN4 was significantly higher than the rest of the groups. This can possibly be explained by the increased surface area available for bonding in combination with the efficacy of the bonding agent applied. The reason for the additional increase of the SIN4 may the tribochemical process of silicatising the surfaces prior to the application of the silane coupling agent. This involves a process were silica-modified aluminum oxide is used to coat the substrate with a thin layer of SiO_2 via sandblasting. The silane molecules react with water to form silanol groups (–Si–OH) from the corresponding methoxy groups (–Si–O–CH_3). The silanol groups then react further to form a siloxane (–Si–O–Si–O–) network with the silica surface thereby increasing the bond energy [11,21,37]. In terms of the mode of failure (Fig. 4), the effect of sandblasting in combination with the bonding agent resulted in all the CER4 specimens failing in mixed mode with no adhesive mode failure indicating the positive effect on the quality of bond compared to the CER2 group, although not significantly stronger. In contrast, the SIG4 group showed an increased proportion of adhesive mode of failure compared to the SIG2 group along with a decrease in bond strength although not significantly different. This would indicate that the effect of the sandblasting had a deleterious effect on the quality of bond contrary to expectation and the authors can offer no explanation for this. In the case of the SIN4 group, this was the only group that showed a proportion of the failure mode to be cohesive within the composite material with the balance, apart from a small proportion of adhesive failure, being mixed mode failure. The cohesive failure mode would indicate that the strength of the bond at the interface was stronger than the intrinsic strength of the composite material and this was reflected in the significantly higher bond strength values for this group.

With regard to identifying mode of failure in dental composite systems bonded to dentine, Scherrer et al. [25] caution the use of a stereomicroscope at low magnification in evaluating the mode of failure and state that this can only be done reliably using SEM. They go on to state that mixed mode and cohesive failure within the composite material cannot be related to a “true” interfacial bond.

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The authors acknowledge this and that our methodology, in using a stereomicroscope to identify the mode of failure, creates a limitation on how much interpretation we can place on the failure process. However, when we consider the purpose of the study was to evaluate the bond between three proprietary composite resin systems and YZr, the mode of failure does indicate whether the bond at the interface is stronger than the intrinsic strength of the composite or not and this information is helpful to inform practitioners when selecting or prescribing materials for prosthodontic restorations. By using a fracture mechanics approach as recommended by Scherrer et al. [25], our expectation was that the crack would propagate along the interface, but the results clearly showed, in the case of the SIN4 group, that the crack, by taking the path of least resistance into the composite material, failed cohesively and therefore the “true” interfacial bond strength was not measured. The same criticism can be made for the mixed mode results.

A relationship was also observed between the elastic modulus of the composite resins (Table 1) and the mean *G*-values. The two materials with the lower elastic moduli (Signum Dentine, *E*-modulus 3.5 GPa; Sinfony Dentine, *E*-modulus 3.1 GPa), recorded higher *G*-values, suggesting the elastic energy build-up in these materials prior to crack initiation were higher than that of the Ceramage Dentine (*E*-modulus 10.7 GPa). This suggests that the lower elastic modulus might result in the material behaving in an elastic manner, where more energy is absorbed thereby requiring a larger energy build-up to initiate failure. The cantilever beam of the specimen underwent deflection as the load was applied. This deflection was caused by the incremental crack growth in the adhesive area between the zirconia plate and the composite cantilever beam [32]. In the case of the Ceramage the higher elastic modulus makes this material more brittle and thus have a lower yielding tolerance than the other two composite materials. Sinfony recorded the highest *G*-values, suggesting that these specimens have greater stored elastic energy that can be converted to specimen surface energy, thereby creating more cracks and rougher surfaces as observed in Fig. 4a [38].

When one compares the range of *G*-values for the composite to YZr bond from this study to those reported for conventional porcelain fused to YZr bonding, a study by Choi et al. reported values ranging between 17.1 Jm^2 and 26.7 Jm^2 , while Li et al. reported values ranging between 10.16 Jm^2 and 18.67 Jm^2 [39,40]. This would indicate that with the appropriate combination of sandblasting and use of a bonding agent with carboxylic acid component (4-META) in combination with an acidic functional monomer containing phosphoric acid (6-MHPA), or alternatively silicatising the YZr surfaces prior to veneering, will produce high strength bonding that exceeds the clinically acceptable bond strength produced in the porcelain fused to YZr systems. As a cautionary note to this, although most of the studies done on the bond between YZr and indirect composite resin have reported a reliable bond between the two materials, several disadvantages of composite materials have been reported in the literature, including insufficient wear resistance, increased plaque accumulation, and surface degradation over time [34].

The authors recognize the small sample size and lack of Weibull analysis as limitations of this study, thus further research with a larger sample size and Weibull analysis is recommended.

5. Conclusion

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

- The application of acidic functional phosphate monomer MDP or silicatising the YZr surfaces before veneering with indirect composite veneering material produced higher bond energy.
- Sandblasting the YZr surfaces with 120 grit $\mathrm{AlO_2}$ only, did not increase the bond energy.

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