J Indian Prosthodont Soc (Jan-Mar 2014) 14(1):38–41
DOI 10.1007/s13191-012-0198-6

ORIGINAL ARTICLE

Evaluation of Shear Bond Strength of Zirconia Bonded to Dentin After Various Surface Treatments of Zirconia

Subash M. Reddy · D. Vijitha · T. Deepak · R. Balasubramanian · A. Satish

Received: 31 July 2012 / Accepted: 16 October 2012 / Published online: 27 October 2012

Abstract Establishing a reliable bond of resin cement to zirconia based materials has been a major limitation against zirconia restoration. Purpose of study: This study evaluated the shear bond strength of zirconia bonded to dentin specimens using a self etch dual cure resin cement after various surface treatments of zirconia. Twenty zirconia rods (3×2.5 mm) were prepared from zirconia blocks and assigned into 5 groups for the following surface treatments. (1) Group I: Control (C) no treatment (2) Group II: airborne-particle abrasion (APA), (3) Group III: hydrofluoric acid etching (HF), (4) Group IV: hydrofluoric acid etching followed by silanation (HF/S), and (5) Group V: application of zirconia primer (Z). Dentin Specimens were prepared from extracted molars stored in 0.5% chloramine-T. Zirconia rods were bonded to dentin using resin cement (Multilink Speed), then light polymerized. The specimens tested for failure were tested using the notched shear bond test method in a universal loading apparatus. One way ANOVA followed by Tukey HSD for cell means were used to analyze the data (=0.05). The highest values were obtained with group V (Z) (8.66 MPa) followed by group II (APA) (6.71 MPa), and group IV (HF/S) (4.41 MPa). The least values were obtained for group III (HF) (3.88 MPa) with no significant difference (*P* value 0.53) between group III (HF) (3.88 MPa) and group I (C) (3.70 MPa). Among the surface treatments employed in this study zirconia primer application is a practical procedure to improved bond strength of zirconia to resin cement followed by Airborne Particle Abrasion with 50 μm Al_2O_3 particles.

Keywords Zirconia · Surface treatment of Zirconia · Bond strength of Zirconia

S. M. Reddy (✉) · D. Vijitha · T. Deepak · R. Balasubramanian · A. Satish
Deptartment of Prosthodontics, Rajah Muthiah Dental College & Hospital, Annamalai University, Chidambaram 608002, Tamil Nadu, India
e-mail: vijithasrinivasan@gmail.com

D. Vijitha
e-mail: vijitha95@gmail.com

A. Satish
e-mail: satishalavandar@gmail.com

Introduction

Increasing esthetic demands has made the pathway for developing metal free materials like zirconia, for fabrication of posterior crowns and fixed partial dentures [1] With the continuing development of the current CAD/CAM systems, dentistry has also became proficient with different zirconia based applications like High strength frameworks, endodontic posts, implants abutments, orthodontic brackets etc.

Zirconium (Zr) is a radio-opaque transition metal element, with a melting point of 1,855 °C and a boiling point of 4,409 °C. It is found in the minerals as Baddeleyite and Zircon ($ZrSiO_4$) and does not exist in a pure state but in conjunction with silicate oxides or as zirconia oxide (ZrO_2). It possesses excellent esthetics, good corrosion resistance, chemical stability, adequate strength and highest room temperature toughness. It is a polymorphic material occurring in 3 forms, monoclinic, tetragonal, and cubic. The monoclinic phase is stable up to 1,170 °C then transforms into tetragonal phase which is stable up to 2,370 °C and the cubic phase is stable up to 2,680 °C, its high strength property is based on the Phase transformation effect [2, 3] This phase transformation is accompanied by volumetric expansion resulting in blunting the propagating crack tips thus increasing the fracture toughness of the material [4].

Zirconia oxide which is stabilized by addition of Yttrium oxide gives a high strength ceramic with better fracture toughness [5]. Thereby making, Yttrium partially stabilized poly crystal material the most preferred zirconia for posterior crowns and FPDs due to its superior biocompatibility, flexural strength and fracture toughness [4]. Its radio opacity enhances radiographic evaluation of marginal integrity, excess cement removal and recurrent decay [6]. Despite the expanded application of zirconia, bonding of zirconia to the resin and the tooth structure is questionable. A good adhesion of resin cement to the tooth structure and the restoration surface is primordial for the success of the ceramic restoration. Several surface treatments like sand blasting, etching with different acids, salinization and primer application is intended to enhance the bond strength by chemical bonding or by micromechanical retention [7].

The objective of this study was to evaluate the shear bond strength of zirconia bonded to dentine specimens using self etch dual cure resin cement, after various mechanical and chemical surface treatments of zirconia (Airborne Particle Abrasion, Hydrofluoric acid etching, Hydrofluoric acid etching followed by silanation and zirconia Primer application).

Materials and Method

Twenty zirconia rods (3.6 mm × 3 mm) were milled from zirconia blocks (Incoris ZI, Sirona) using CAD/CAM (Sirona dental systems, GmbH) and sintered at 1,500 °C for 7 h (Sintramat; Ivoclar Vivadent AG). One side of each rod was finished manually using 600 grit silicon carbide paper to make a flat surface. The rods were then divided into five groups of four rods in each group (Fig. 1). The sample was mounted separately in a light body vinyl polysiloxane material (Zeta plus; Zhermack) and subjected to the surface treatments.

- Group I (C) was the control group where no surface treatment was done.
- Group II (APA) zirconia rods were subjected to Airborne particle abrasion with 50 µm Al_2O_3 particles at 3.0 bar pressure from a distance of 10 mm parallel and perpendicular to the long axis of the bars, for 10 s.
- Group III (HF) zirconia rods were subjected to hydrofluoric acid 4.5 % etching (IPS Ceramic etching gel; Ivoclar Vivadent AG) for 3 min and then dried.
- Group IV (HF/S) zirconia rods were subjected to hydrofluoric acid 4.5 % etching (IPS Ceramic etching gel; Ivoclar Vivadent AG) for 3 min and silanated by application of a silane coupling agent (Monobond-S; Ivoclar Vivadent AG) for 2 min, then dried.

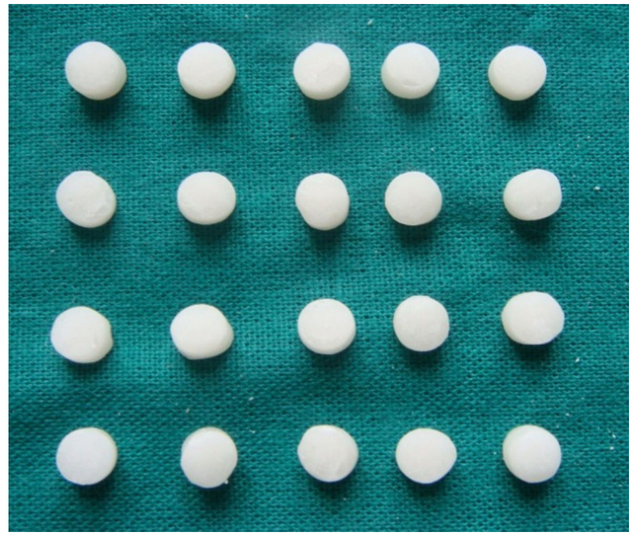

Fig. 1 Zirconia bars divided into five groups of four rods in each group

- Group V (Z) zirconia rods were subjected to application of zirconia primer (Metal/Zirconia primer; Ivoclar Vivadent AG) (Fig. 2) on intaglio surfaces of the rods for 3 min, then dried.

Twenty extracted permanent molars were collected and stored in 0.5 % Chloramine T (Explicit Chemicals Pvt Ltd, Pune) for 1 week. The buccal and lingual surfaces of each molar were ground flat using 600 grit Silicon carbide paper to expose dentin. Each tooth was sectioned longitudinally in a mesiodistal direction using a precision saw with a high concentration diamond watering blade underwater cooling. Tooth sections were mounted in auto polymerizing acrylic resin (DPI Cold cure) measuring 25 mm in diameter and 26 mm in height (Fig. 3) and were assigned into 5 groups ($n = 4$). Each group was assigned one of the pretreated zirconia rod groups. The exposed dentin surface was finished with 600 grit 8 inch grinding disks, rinsed with water, and gently air dried. A dual polymerizing self etching adhesive

Fig. 2 Zirconia primer used for surface treatment of Group V

Fig. 3 Tooth section mounted in auto polymerizing acrylic resin

Fig. 4 Zirconia rod bonded to dentin specimens using dual polymerizing self etching adhesive cement

cement (Multilink Speed, Ivoclar Vivadent AG) was used to bond the zirconia rods to dentin specimens according to manufacturer's instructions (Fig. 4). Excess cement was removed, and the margins were light polymerized using a halogen polymerization light (3 M ESPE) at 1,350 mw/cm^2 for 20 s on either sides of the specimens. The specimens were stressed with notched shear bond test using an universal loading apparatus (Unitek 94100, FIE) setup for compression testing using a 0.05 kN load cell at 1 mm/min cross head speed (Fig. 5).

Statistical Analysis

One way ANOVA followed by Tukey HSD for cell means were used to analyze the data ($\alpha = 0.05$), with mechanical and chemical treatments as independent variable.

Results

All specimens showed failure at zirconia resin interface. ANOVA showed significant differences in bond strength among the different surface treatments with the *P* value 0.001. The highest values were obtained with group V (Z) (8.66 MPa) followed by group II (APA) (6.71 MPa) and group IV (HF/S) (4.41 MPa). There were no significant difference between group III (HF) (3.88 MPa) and group I (C) (3.70 MPa) (Table 1).

Discussion

Zirconia based ceramics are gaining popularity because of their superior esthetics, high flexural strength, a relatively low elastic modulus, and a high fracture toughness compared to other ceramic materials [8] Yttria stabilized tetragonal zirconia (Y-TZP) has opened new vistas for all

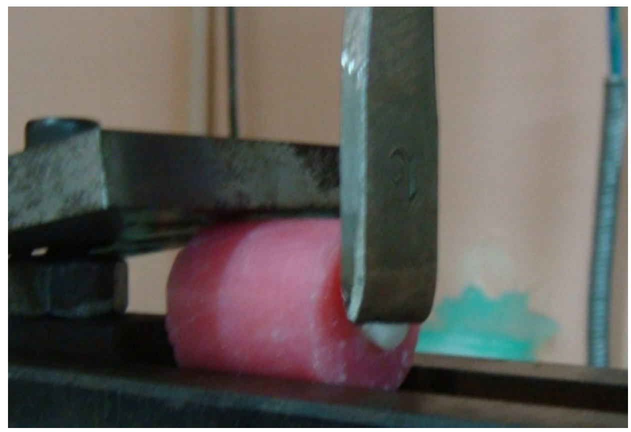

Fig. 5 The specimens were stressed with notched shear bond test method using an universal loading apparatus

ceramic FPD even in loaded reconstructions like the molar region [9].

Heather [10] stated that the strength of an all ceramic restoration is dependent on the ceramic material used, core veneer bond strength, crown thickness, and design of the restoration, as well as bonding techniques. Previous

Table 1 The recorded values of stress for various surface treated samples

Group	No. of samples	Mean	SD	F-value	*P* value
I (C)	4	3.70	0.35	129.52	0.001 Significant
II (APA)	4	6.71	0.40		
III (HF)	4	3.88	0.40		
IV (HF/S)	4	4.41	0.35		
V (Z)	4	8.66	0.35		

Group I: (C) Control Group, Group II: (APA) Airborne particle abrasion, Group III: (HF) Hydrofluoric acid etched, Group IV: (HF/S) Hydrofluoric acid etched and silanated, Group V: (Z) Zirconia primer treated investigations revealed that most clinical failures ranging between 2.3 and 8 % are due to cementation or internal surfaces [11] The integrity of the luting cement to ceramic surfaces plays a major role in the longevity of the restoration identified the need for a reliable conditioning method like surface treatment to strengthen this critical area [12].The surface treatments available are of two types namely mechanical and chemical surface treatments. In this study the various surface treatments evaluated were Airborne Particle Abrasion, Hydrofluoric acid etching, Hydrofluoric acid etching followed by silanation and Zirconia Primer application.

In this study, The group III (HF) attained the least bond strength value (3.88 MPa) among surface treated groups with no significant difference from the control group (C) (3.70 MPa) similar to the results concluded by Bottino et al. [13]. Torres [14] evaluated the effect of acid etching on zirconia by SEM analysis and reported that the surface morphology was not changed by HF acid etching. Thus, Hydrofluoric acid etching is not an effective method in glass free zirconia oxide ceramics as this material is resistant to acidic and alkaline corrosive material [4].

The group IV (HF/S) attained a bond strength value of 4.41 MPa Etching the inner surfaces of ceramics using Hydrofluoric acid followed by the application silane coupling agent does not produce a chemical bond, but may improve the wetting ability of the surface resulting in small but increased bond strength value [12, 15, 16].

The group II (APA) achieved a bond strength value of 6.71 MPa, This was in agreement with a study by Qublewi [17]. APA removes loose contaminated layers and the roughened surface provides some degree of mechanical interlocking or keying with the adhesive. It can be due to the fact that the increased roughness has also increased the surface area for the bonding to occur [12].

Metal/zirconia Primer is a single-component priming agent designed to mediate an optimal chemical bond between metal alloys or ceramic oxide to methacrylate-based luting composite. The group V (Z) achieved the maximum bond strength of 8.66 MPa which is agreement with the study by Qublewi et al. [17] Aboushelib et al. [4], Esam [18] .

Conclusion

Within the limitations of this in vitro study it is conclusive of the facts that.

1. Surface treatment of zirconia can improve its the bond strength to the resin.
2. Among the surface treatments employed in this study zirconia primer application is a practical procedure to improved bond strength of zirconia to resin cement followed by Airborne Particle Abrasion with 50 µm Al_2O_3 particles .

References

1. Denrya Isabelle, Kelly JR (2008) State of the art of zirconia for dental applications. Dent Mater 24:299–307
2. Garvie RC, Hannink RH, Pascoe RT (1975) Ceramic steel? Nature 258:703–704
3. Piconi C, Maccauro G (1999) Zirconia as a ceramic biomaterial. Biomaterials 20:1–25
4. Aboushelib MN, de Jager N, Kleverlaan CJ (2006) Microtensile bond strength of different components of core veneered all—ceramic restorations. Part II. Dent Mater 22:857–863
5. Derand T, Molin M, Kvam K (2005) Bond strength of composite luting cement to zirconia ceramic surfaces. Dent Mater 21:1158–1162
6. Raigrodski AJ (2004) Contemporary all ceramic fixed partial denture : a rewiew. Dental Clin North Am 48:531–544
7. Stangel I, Nathanson D, Hsu CS (1987) Shear strength of composite bond to etched porcelain. J Dent Res 66:1460–1465
8. Evans AG (1990) Perspective on the development of high-toughness ceramics. J Am Ceram Soc 73:187–206
9. Fischer J, Grohmann P, Stawarcyzki B (2008) Effect of zirconia surface treatments on the shear strength of zirconia/veneering ceramic composites. Dent Mater J 27(3):448–454
10. Heather J, Wook J, Pesun IJ (2007) Current ceramic materials and systems with clinical recommendations: a systematic review. J Prosthet Dent 98:389–404
11. Libby G, Rrcuri MR, La Velle WE (1997) Longevity of fixed partial dentures. J Prosthet Dent 78:127–131
12. Amaral R, Ozcan M, Bottino MA, Valandro LF (2006) Micro tensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: the effect of surface conditioning. Dent Mater 22:283–290
13. Bottino MA, Valandro LF, Scotti R (2005) Effect of surface treatments on the resin bond to Zirconium-based ceramics. Int J Prosthodont 18:60–65
14. Torres SMP, Borges GA, Spohr AM (2009) The effect of surface treatments on the micro-shear bond strength of a resin luting agent and four all-ceramic systems. Oper Dent 34(4):399–407
15. Kern M, Blatz MB, Sadan A (2003) Resin-ceramic bonding: a review of the literature. J Prosthet Dent 89:268–274
16. Madani M, Chu FC, McDonald AV (2000) Effect of surface treatments on shear bond strength between a resin cement and an alumina core. J Prosthet Dent 83:644–647
17. Qeblawi DM, Munoz CA, Brewer JD, Monaco EA Jr (2010) The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. J Prosthet Dent 103:210–220
18. Esam T (2009) Effect of surface treatment on the micro-shear bond strength to zirconia. Saud Dent J 21:113–116