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Bond strength of resin cement to zirconia treated in pre-sintered stage

Kamal Ebeid^{a,b,*}, Sebastian Wille^a, Tarek Salah^b, Marwa Wahsh^b, Maged Zohdy^b, Matthias Kern^a^a Department of Prosthodontics, Propaedeutics and Dental Materials, School of Dentistry, Christian-Albrechts University at Kiel, Germany^b Department of Fixed Prosthodontics, Faculty of Dentistry, Ain Shams University, Egypt

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ABSTRACT

Purpose: Aim of this study was to evaluate the tensile bond strength (TBS) between resin cement and zirconia surface treated in different sintering stages.

Materials and methods: Eighty zirconia discs having final dimensions of 12 mm diameter and 3.2 mm thickness were milled then divided into three main groups according to the type of surface treatment performed (group 1: air abrasion using 50 μm Al₂O₃ particles, group 2: silica coating using Rocatec soft, and group 3: a control group receiving no surface treatment). Groups 1 and 2 were divided into two subgroups each according to the stage in which the surface treatment was performed (subgroup A; surface treatment performed in the pre-sintered stage and subgroup B; surface treatment performed in the post-sintered stage). Discs were later bonded to composite core materials using resin cement then tested for TBS either being subjected to 3 days of water storage or 150 days of water storage and 37,500 thermal cycles.

Results: Long-term aging caused a significant decrease in TBS of all subgroups except the subgroup air-abraded in the post-sintered stage. After long-term aging, the group silica coated in the pre-sintered stage showed the significantly lowest TBS compared to all other groups. There was also no significant difference between the subgroups air-abraded and silica coated in the pre-sintered stage. All specimens in the control group debonded during long-term aging.

Significance: Air-abrading zirconia with Al₂O₃ at a reduced pressure in the pre-sintered stage may result in durable bond strength to resin cement.

1. Introduction

The development of advanced dental ceramics has led to the application of densely sintered zirconia in restorative dentistry which can be produced using computer aided design/computer-aided manufacturing (CAD/CAM) systems. The use of zirconia ceramics for dental restorations became more popular due to their superior fracture strength and toughness when compared to silica-based dental ceramics (Tinschert et al., 2000, 2001; Guazzato et al., 2004). In addition to its high mechanical strength zirconia ceramic exhibits exceptional biocompatibility (Piconi and Maccauro, 1999; Manicone et al., 2007).

When zirconia ceramic restorations with little or no mechanical retention are used, their clinical outcome is highly dependent on the establishment of a strong adhesion between the luting cement and zirconia. Compared with traditional luting cements, resin cement has some irreplaceable advantages including higher mechanical strength and better esthetic properties (Piwowarczyk et al., 2005). However, without any surface treatment, the resin zirconia bonding was found to be susceptible to aging conditions (Özcan et al., 2008). Traditional

ceramic bonding methods, such as acid etching followed by the application of silane coupling agents, could not effectively improve bonding to zirconia due to the chemical inertness of zirconia and the absence of a glass phase (Denry and Kelly, 2008; Ho and Matinlinna, 2011). Thus, zirconia ceramics cannot be etched with commonly used acids, such as hydrofluoric and phosphoric acids for increasing surface roughness. Furthermore, silane coupling agents do not promote chemical bonding to zirconia, as it contains no silica (Kern and Wegner 1998; Özcan and Vallittu 2003; Komine et al., 2012).

Air-abrasion with alumina particles followed by an appropriate chemical bonding process was recommended to achieve long-term retention to zirconia (Kern and Wegner, 1998). The incorporation of 10-methacryloxydecyl dihydrogen phosphate (MDP) in resin cements and in primers was a major factor in producing durable resin zirconia bonding which has already been confirmed in the related clinical trials (Abou Tara et al., 2011; Sasse and Kern, 2013). Other surface treatments, such as the tribochemical silica coating, selective infiltration etching, heating with a hot etching solution, laser surface treatment, plasma treatment and surface fluorination, have been developed to

* Corresponding author at: Department of Prosthodontics, Propaedeutics and Dental Materials, School of Dentistry, Christian-Albrechts University at Kiel, Germany.
E-mail address: kebeid@proth.uni-kiel.de (K. Ebeid).

enhance bonding to zirconia (Heikkinen et al., 2009). The tribochemical silica coating method has been proven not only to increase the values of surface roughness, but also to add silica content on zirconia surface. Silica surface content is a precondition for the function of silane coupling agents. Both mechanical interlocking and chemical bonding between resin cement and zirconia have thus been enhanced (Peutzfeldt and Asmussen, 1988).

However, there are still concerns about the influence of air-abrasion and tribochemical silica coating on the mechanical properties and long-term stability of zirconia ceramic since it has been reported that they induce some flaws and phase transformation on the surface, thus promoting low temperature degradation (Zhang et al., 2004). The generation of such flaws and transformation might produce some detrimental effects on the liability of zirconia ceramic (Kosmac et al., 1999; De Souza et al., 2016). There are also some concerns about the durability of the bond strength to silica-coated zirconia ceramic as many laboratory studies showed that it decreased significantly after a few months of artificial aging (Kern and Wegner, 1998; Wegner and Kern, 2000; Blatz et al. 2003; Özcan and Vallittu 2003; da Silva et al., 2014).

Although several systematic reviews discussed the variety of tested surface treatments and their reliability, none of them revealed any studies evaluating bonding to surface treated zirconia in the pre-sintered stage (Kern, 2009, 2015; Thompson et al., 2011; Inokoshi et al., 2014; Papia et al., 2014; Ozcan and Bernasconi, 2015).

Thus the aim of this study was to evaluate the effect of air-abrasion and silica coating of zirconia in different sintering stages on the tensile bond strength of resin cement. The hypotheses of this study were that the type of surface treatment and the stage in which it is performed will not affect the tensile bond strength of resin cement to zirconia.

2. Materials and methods

Eighty zirconia ceramic discs (Bruxzir, Glidewell, California, USA) having final dimensions of 12 mm diameter and 3.2 mm thickness were milled using a dental milling machine, polished using SiC paper till 600 grits then divided into three main groups according to the type of surface treatment performed (group 1; (n = 32) air-abrasion using 50 µm Al₂O₃ particles, group 2; (n = 32) silica coating using Rocatec soft (3M ESPE, St Paul, USA), and group 3; (n = 16) control group receiving no surface treatment or polishing). Groups 1 and 2 were divided into two subgroups each according to the stage in which the surface treatment was performed (subgroup A; surface treatment performed in the pre-sintered stage and subgroup B; surface treatment performed in the post-sintered stage). For all surface treatments particle abrasion was performed 15 s from a distance of 10 mm in a crosswise motion. In subgroup A air-abrasion with alumina was applied with 0.5 bars pressure, while in subgroup B for silica-coating an air pressure of 2.8 bars was used. Specimens were later ultrasonically cleaned in 99% isopropanol for 3 mins and then dried with air.

From each subgroup the treated surface of a sample was investigated by x-ray photoelectron spectroscopy (XPS). For these measurements an Omicron Full Lab (Omicron NanoTechnology GmbH, Taunusstein, Germany) with an Al K α X-ray source and a VSW 100 hemispherical analyzer was used. Quantification of the surface silica content was calculated using the software CasaXPS Version 2.3.17.

Transparent plastic tubes with 3.2 mm inner diameter were filled with freshly mixed composite resin (Clearafil DC core plus, Kuraray, Osaka, Japan) and allowed to polymerize for 7 mins in an incubator at 37 °C before bonding. Zirconia specimens were primed using a universal primer (Clearafil ceramic primer plus, Kuraray) and bonded to the resin filled tubes with a dual-curing luting resin (Panavia V5, Kuraray) using an alignment apparatus under a load of 750 g. The specimens bonded were light-cured for 20 s from two opposite sides with a dental curing light device (Optilux 500, Kerr, Danbury, USA). For each subgroup eight randomly selected specimens were stored in a distilled water bath at 37 °C for 3 days without thermal cycling to record the initial bond

strength. The other 8 specimens were stored for 150 days in the 37 °C water bath, interrupted by thermal cycling between 5 °C and 55 °C in distilled water with a dwell time of 30 s (Willytec, Munich, Germany) for 37,500 cycles. After the different storage conditions, tensile bond strength (TBS) was tested with a universal testing apparatus (Zwick Z010, Ulm, Germany) at a crosshead speed of 2 mm/min using a self-aligning chain loop attachment.

The fractured interfaces of the zirconia ceramic specimens were examined with a light microscope (Zeiss S7, Carl Zeiss, Oberkochen, Germany) at 20 × magnifications to assess the failure modes. Debonded surfaces were assigned to cohesive failure within luting resin or composite resin, adhesive at ceramic/cement interface or mixed adhesive/cohesive modes. Failure areas of each mode were calculated and expressed as a percentage of the total bonding surface area for each test group. Representative samples were examined in a scanning electron microscope (SEM, XL 30 CP, Philips, Kassel, Germany) with an acceleration voltage of 15 KeV after sputtering using a gold alloy conductive layer of approximately 30 nm.

The data collected was checked for normal distribution using Shapiro-Wilk test and analyzed using two-way and three-way analysis of variance (ANOVA), followed by Tukey's HSD test (SPSS v20, Chicago, IL, USA) at a significance level of $P \leq 0.05$.

3. Results

XPS revealed that specimen silica coated before sintering showed a 5.62% of silica before sintering that decreased to 2.02% after sintering. XPS measurements also showed that air-abraded groups and the not treated control group showed no silica content, while the group silica coated in post-sintered stage showed 19.68% of silica.

Three-way ANOVA revealed statistically significant differences between subgroups. Significant differences were found between the stages of the surface treatment, between the surface treatments itself, and with and without thermal cycling. A significant interaction was found between the three variables. Thermal cycling caused a significant decrease in TBS of all subgroups except the subgroup air-abraded in the post-sintered stage. After thermal cycling, TBS of subgroups did not differ significantly except for the group silica-coated in the pre-sintered stage, which showed the lowest TBS. There was also no significant difference between the subgroups air-abraded and silica-coated in the pre-sintered stage. Regarding the control group all specimens debonded during thermal cycling (Table 1).

Further analysis using two-way ANOVA was conducted to analyze the initial bond strength. Statistical analysis revealed statistically significant differences between subgroups. Significant differences were found between the stages of the surface treatment and between the surface treatments itself. Significant interaction was found between the stage and type of the surface treatment. Subgroups treated in the post-sintered stage showed higher TBS when compared to the subgroups treated in the pre-sintered stage and the control group. Regarding surface treatments, statistical significance was found between silica coating and air-abrasion only in the group treated in the post-sintered stage. All subgroups showed higher TBS when compared to the control group.

As for the specimen's mode of failure, specimens tested after 3 days showed a predominantly cohesive failure except for the control group, which showed a pure adhesive failure. After thermal cycling silica-coated specimens showed a shift from cohesive to adhesive failure, while air-abraded specimens still had a predominantly cohesive mode of failure. The control group debonded spontaneously during thermal cycling with a pure adhesive mode of failure (Figs. 1–6).

4. Discussion

For surface treatments done in pre-sintered stage, a pilot study was conducted in order to determine the optimum pressure. Zirconia discs

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