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Bond Strength of Resin Cements to Zirconia after Surface Treatments

Mehari K, Parke A, Gallardo F, Vandewalle KS

Abstract

Air-abrasion with glass beads may reportedly be less likely to weaken cubic-containing zirconia compared to air abrasion with aluminum oxide. However, the effect of air-abrasion with glass beads on the bond strength of resin cement to cubic-containing zirconia is less known. **Objective:** The purpose of this study was to evaluate the effects of air abrasion with aluminum oxide or glass beads on the shear bond strength of resin cement to three types of zirconia containing various levels of cubic crystalline phases (3Y-TZP, Katana ML; 4Y- PSZ, Katana STML; and 5Y- PSZ, Katana UTML, Noritake). **Methods:** Thirty block specimens (8x8x3.5mm) were milled out of each zirconia material and mounted in plastic pipe. Ten specimens of each of the zirconia materials were air-abraded using 50µm aluminum-oxide particles; ten specimens were abraded using 80 µm glass beads; and ten specimens served as a control and received no surface treatment. A 10-MDP primer was applied to the surface of the zirconia specimens. Composite discs were bonded using a resin cement and light cured. The specimens were stored in 37°C distilled water for 24 hours and thermocycled for 2,500 cycles. The specimens were loaded in shear on a universal testing machine. Data were analyzed with one- and two-way ANOVAs and Tukey post hoc tests (alpha=0.05). **Results:** A significant difference in shear bond strength was found based on surface treatment ($p<0.001$), but not on type of zirconia ($p=0.132$). **Conclusions:** Air-abrasion with glass beads or no surface treatment resulted in significantly lower bond strength of the resin cement to all three zirconia types compared to air-abrasion with aluminum oxide.

Introduction

Zirconia is a metastable material that can exist in various crystalline phases. Three types of which have been utilized for dentistry: monoclinic, tetragonal, and cubic. The first version of zirconia employed in dentistry was a form comprised of the high-strength tetragonal crystalline phase.[1] At room temperature, zirconia exists in the weaker monoclinic crystalline form. However small amounts of oxides, or dopants like yttrium oxide (Y_2O_3) are added to stabilize it in the tetragonal crystalline form.[1] Y_2O_3 is added to the purified zirconia powder to stabilize the tetragonal phase and prevent it from transforming to the weaker monoclinic phase with sintering.[1] Of all the restorative ceramics, Y_2O_3 stabilized tetragonal zirconia polycrystal (Y-TZP) is the most clinically durable.[2]

However, current Y-TZP tetragonal zirconia materials on the market lack the aesthetics of competitive glass-ceramics and are therefore somewhat restricted to the posterior region or frameworks.[2] To increase translucency, new formulations of zirconia oxide were developed. Formulating new translucent dental zirconia materials involved increasing the Y_2O_3 content, which introduced the cubic phase along with the metastable tetragonal phase in the resulting zirconia-oxide materials.[3] The presence of specific percentages of tetragonal and cubic phases defines the mechanical and physical properties of the zirconia. Using a higher Y_2O_3 content produces a partially stabilized zirconia (PSZ) with greater cubic content. For example, the amount of Y_2O_3 dopant in molar concentration that is used in zirconia is abbreviated as 3Y-TZP for 3 mol% Y_2O_3, 4Y-PSZ for 4 mol% Y_2O_3, or 5Y-PSZ for 5 mol% Y_2O_3.[3] In these new zirconia materials, the quantity of the cubic phase increases from 15% in 3Y-TZP materials to approximately 25 vol% in 4Y-PSZ materials and up to 50 vol% in 5Y-PSZ materials. This increased cubic phase improves the translucency of the zirconia materials compared to traditional 3Y-TZP zirconia materials but diminished toughness and strength.[4] 3Y-TZP has a high fracture toughness from 5 to 10 MPa·m½ and a flexural strength of 900 to 1400 MPa.[3] 4Y-PSZ has a fracture toughness of 2.5 to 3.5 MPa·m½ and flexural strength of 600 to 900 MPa, and 5Y-PSZ has a fracture toughness of 2.2 to 4 MPa·m½ and flexural strength of 700 to 800 MPa.[4]

One of the unique things about zirconia is its increased ability to resist cracks. This property is called transformation toughness. The transformation of zirconia's metastable tetragonal phase to the monoclinic phase allows for this toughening, which increases a zirconia restoration's resistance to fracture.[5] A certain disadvantage of 4Y-PSZ and 5Y-PSZ is the lower fracture toughness compared to 3Y-TZP. The translucent materials have smaller amounts of tetragonal phase (75% in 4Y-PSZ and ~50% in 5Y-PSZ), leading to a reduced possibility of tetragonal to monoclinic transformation and therefore less transformation toughening.[4]

Zirconia restorations are considered cementable with conventional cements due to their high flexural strength, but may need to be bonded if mechanical retention is limited.[6] Bonding to 3Y-TZP has been studied with recommendations that alumina-oxide abrasion and adhesive monomer application be utilized for more predictable long-term bonding. Concerns have been raised regarding surface damage after air abrading 3Y-TZP with aluminum oxide particles.[7,8] However, the results from a subgroup analysis demonstrated that air abrasion may actually improve the mechanical strength of 3Y-TZP compared to non-abraded specimen, irrespective of the air pressure or duration.[9] No strength decrease was observed even after longer abrasion times.[7] The monomer 10-methacryloyloxy-decyl dihydrogenphosphate (10-MDP) was originally designed to bond to metal oxides and its use has been extended to oxide ceramics. 10-MDP-containing resin cements or primers seem to be the most successful due to the chemical interaction between the hydroxyl groups of the passive zirconia surface and the phosphate ester group of the 10-MDP.[10] A recent study examined the bond strength between an adhesive resin cement and cubic-containing zirconia and found it was similar to traditional tetragonal zirconia.[11] However, the weaker cubic-containing zirconia materials may require a different bonding strategy when using air abrasion.

Cubic-containing zirconia may not undergo transformation toughening and therefore, may be more susceptible to mechanical damage with aluminum-oxide air abrasion. The surface treatment with glass beads instead of aluminum oxide has been suggested for cubic-containing zirconia, and does not seem to result in a strength degradation.[12] A recent study found that the flexural strength of esthetic cubic-containing zirconia was adversely affected by aluminum-oxide air abrasion and to a lesser extent by glass beads.[13] Although glass beads maybe less likely to weaken the cubic-containing zirconia, it may not prepare the surface as well as aluminum oxide and therefore, it may result in lower bond strength of resin cement.

The purpose of this study was to evaluate the effects of aluminum oxide and glass bead air abrasion on the shear bond strength of resin cement to 3Y-TZP, 4Y-PSZ and 5Y-PSZ zirconia materials. The null hypothesis tested was that there would be no difference in shear bond strength of a resin cement based on (1) surface treatment or (2) type of zirconia.

Methods and Materials

The following materials were tested: 3Y-TZP Multi-Layered (Katana ML, Shade 1.5-2), 4Y-PSZ Super-Translucent Multi-layered (Katana STML, Shade A2) and 5Y-PSZ Ultra-Translucent Multi-layered Zirconia (Katana UTML, Shade A2, Kuraray Dental, Houston, TX, USA). Aluminum-oxide particles and glass beads were used for air-particle surface treatment. Ninety specimens were created for shear bond strength testing. Ten specimens per each of the three ceramic materials were created and subjected to three different surface treatments (aluminum-oxide air abrasion, glass-bead air abrasion or no surface treatment).

Thirty block specimens (8x8x3.5mm) were designed and milled out of each of the zirconia materials. The blocks were designed using DS SolidWorks software (SolidWorks, Waltham, MA, USA) and the file was imported into Sum 3D, iCAM V5 milling software (I-Mes, iCore, Eiterfeld, Germany). A CAM (computer-aided manufacturing) machine (I-Mes iCore 450i) was used to mill the zirconia blocks out of the zirconia blanks. After sintering the blocks in a furnace (Programat S1 1600, Ivoclar Vivadent, Amherst, NY, USA), the zirconia blocks were mounted in plastic pipe using resin (Vitacrilic, Fricke, International, Streamwood, IL, USA). The top surface of the block specimen received either air abrasion with aluminum oxide, glass beads, or no surface treatment. The blocks from the aluminum-oxide group were air-abraded (Basic Quattro IS, Renfert, Chicago, IL, USA) using 50 μm aluminum oxide (Korox, BEGO, Bremen, Germany) at 2.0 bar at a distance of 10mm for 10 seconds using a vinyl polysiloxane jig to standardize distances. Similarly, the blocks from the glass-bead group were air-abraded using 80 μm glass beads (Perlablast Micro, BEGO, Bremen, Germany). A control group received no surface treatment. Then the blocks were steam cleaned (i700B, Reliable, Toronto, Ontario).

Ninety composite resin discs (Z250 3M ESPE, St Paul, MN, USA) were produced using a custom-made metal split mold (4.0-mm internal diameter and 2.0-mm thickness) positioned between two glass slabs covered with transparent polyester films. Light polymerization of the composite were performed for 40 seconds on the top surface and two diametrically opposed sides of the resin discs after removal from the mold (total of

120 seconds) using the Bluephase 20i (Ivoclar Vivadent) light-curing unit. Irradiance of the curing light was determined with a radiometer (LED Radiometer, Kerr, Orange, CA, USA) to verify irradiance levels of at least 1200 mW/cm^2. A thin layer of phosphate-based 10-MDP primer (Z-Prime Plus, Bisco, Schaumburg, IL, USA) was applied to the top surface of the zirconia block according to the manufacturer's instructions. A dual-cure resin cement (NX3, Kerr) was used to bond to the prepared composite cylinders to the zirconia blocks. A thin layer of the mixed cement was applied and distributed to the bonding surface. A 100-gram calibration weight was placed on the composite cylinders to ensure a standardized film thickness of cement. After excess cement was removed, the cement was cured in four equidistant positions for 40 seconds each using the curing light as before.

The specimens were stored in 37°C distilled water in a lab oven (Model 20GC, Quincy Lab, Chicago, IL, USA) for 24 hours and then thermocycled in distilled water for 2500 cycles at 5°C and 55°C with a dwell time of 30 seconds at each temperature (Sabri Dental Enterprise, Downers Grove, IL, USA). The specimens were loaded perpendicularly with a blade-shaped probe in a universal testing machine (Model 5943, Instron, Norwood, MA, USA) using a crosshead speed of 1.0 mm/min until failure. Shear bond strength values in megapascals (MPa) was calculated from the peak load of failure divided by the specimen surface area. A mean and standard deviation were determined per group. The data were submitted first to a Shapiro Wilk test and found to be normally distributed. The data were then analyzed with a two-way ANOVA and Tukey post hoc test evaluating the effect of zirconia material (3-levels) or surface treatment (3-levels) on shear bond strength (alpha = 0.05). Additionally, the data were analyzed with multiple one-way ANOVAs per zirconia material or surface treatment.

Following testing, each shear bond strength specimen was examined using a light microscope (SMZ-1B, Nikon, Melville, NY, USA) at 10x magnification to determine failure mode as either: 1) adhesive fracture at the resin cement/zirconia interface, 2) cohesive fracture in resin cement, 3) mixed (combined adhesive and cohesive) in resin cement or zirconia, or 4) cohesive fracture in zirconia. Representative specimens from each group were imaged with a scanning electron microscope (TM 3000, Hitachi, Tarrytown, NY) at

40x magnification. Statistical analysis was performed using SPSS 25 (IBM/SPSS, Chicago, IL, USA).

Results

A significant difference in shear bond strength of the resin cement was found based on surface treatment ($p<0.001$), but not on zirconia type ($p=0.132$), with no significant interaction ($p=0.98$). Air abrasion with aluminum oxide created significantly greater bond strength of the resin cement to all zirconia materials compared to air abrasion with glass beads or no surface treatment. No significant difference in shear bond strength of the resin cement was found between the use of glass beads and no surface treatment (see Table 1). There was no significant difference in shear bond strength between any of the three zirconia materials based on surface treatment. More mixed failures were observed with aluminum oxide air abrasion (see Figure 1). Representative failure modes at 40x magnification are shown in Figure 2.

Discussion

The survival of zirconia restorations with less retentive preparations relies on, among other aspects, the durability of the bonded interfaces.[2] Zirconia is a densely sintered material exhibiting high hardness and thus roughening the surface of the ceramic may be more difficult.[14,15] Zirconia does not contain silica-glass particles like traditional glass ceramics allowing an etch with hydrofluoric acid and bond with silane to the intaglio surface. Therefore it may require additional surface treatment (air abrasion) and surface primers for less retentive preparations. Air-abrasion may clean the ceramic surface, remove impurities, and increase surface roughness, bond strength, surface energy and wettability.[2] Air abrasion has been reported to both increase and decrease the mechanical strength of 3Y-TZP zirconia materials and to promote varied percentages of phase transformation.[16-18] These contradictory findings may result from the diverse protocols used, with variation in particle size and pressure, as well as the lack of aging conditions. However, global results from a meta-analysis showed that the use of surface conditioning procedures based on airborne-particle abrasion protocols improved the flexural strength of 3Y-TZP.[7] The force of the particles on the 3Y-TZP surface can lead to microcracks and plastic deformation,[16,19] thus decreasing the strength of the ceramic. However, small cracks, flaws, and defects that could lead to fracture appear to remain confined within the transformation layer, where they are probably healed by the 4% volume increase in the grains during the phase transformation.[20] However, a recent study found that the flexural strength of 4Y-PSZ and 5Y-PSZ cubic-containing zirconia materials were adversely affected by aluminum-oxide air abrasion and to a lesser extent by the use of glass beads.[13]

In this study, air abrasion with aluminum oxide created greater bond strength of the resin cement to all zirconia materials compared to air abrasion with glass beads or no surface treatment. Therefore, the first null hypothesis was rejected. The use of the glass beads resulted in a zirconia surface that was similar in retention to no surface treatment but less retentive than the use of the aluminum-oxide particles. Although glass beads maybe less likely to weaken the cubic-containing zirconia,[13] the results of this study suggest it may not prepare the surface as well as aluminum oxide and therefore lower the bond strength to resin cement. Similar to this study, a recent study by Le et al. found that aluminum-oxide air abrasion increased the bond strength significantly for both conventional and translucent zirconia.[11]

Although there were differences in cubic content and inherent strength properties of the three materials, there was not a significant difference in bond strength per surface treatment. Therefore, the second null hypothesis was not rejected. Air abrasion with aluminum oxide resulted in over twice the shear bond strength than the use of glass beads or no surface treatment - irrespective of material type. Additionally, more mixed failures were associated with aluminum-oxide air abrasion compared to the use of glass beads. Mixed failure modes are often associated with higher bond strength compared to purely adhesive type failures, as was seen more readily with the use of glass beads and even more evident in the untreated control group.[21] Limitations to this study include the use of only one brand of zirconia, cement, primer, and particle size and pressure.

Conclusions

Air-abrasion with glass beads or no surface treatment resulted in significantly lower bond strength of the resin cement to all three zirconia types compared to air-abrasion with aluminum oxide.

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Table 1. Shear bond strength of the resin cement to various zirconia types after surface treatments with no treatment (control), glass beads or aluminum oxide. Groups with the same upper case letter per row or lower case letter per column are not significantly different (p>0.05)

Surface Treatment	**Shear Bond Strength** MPa (st dev)		
	Katana ML	**Katana STML**	**Katana UTML**
Control	6.0 (1.5) Aa	5.1 (1.3) Aa	6.4 (1.4) Aa
Glass Beads	5.9 (1.7) Aa	4.8 (1.0) Aa	5.6 (1.1) Aa
Al Oxide	13.4 (3.8) Ab	11.8 (4.1) Ab	12.9 (3.4) Ab

Figure 1: Failure modes of the resin cement to the various zirconia types after surface treatment with no treatment (control), glass beads or aluminum oxide. Failures were found to be adhesive or mixed and expressed as a percentage of all specimens per group.

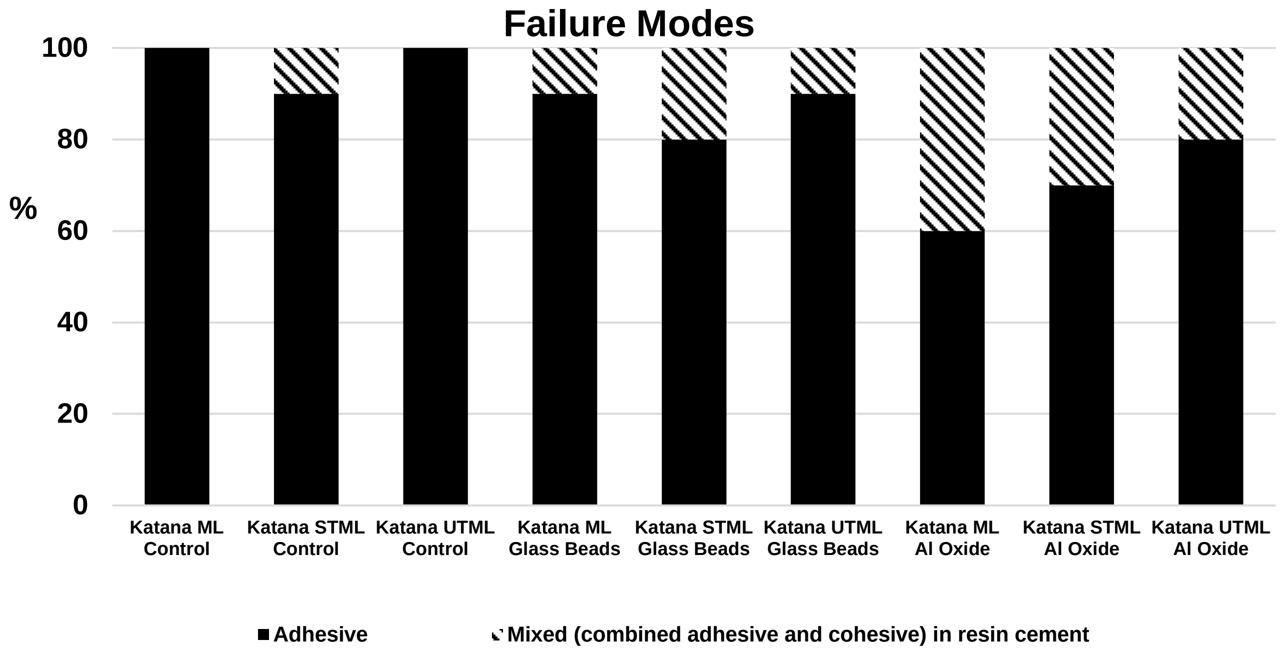

Figure 2: Representative failure modes at 40x magnification. (A) Primary failure through the adhesive interface as seen with many of the control (no surface treatment) and air abrasion with glass bead specimens. (B) Mixed fracture through the adhesive layer and cement was seen more commonly with the aluminum oxide air-abrasion specimens.

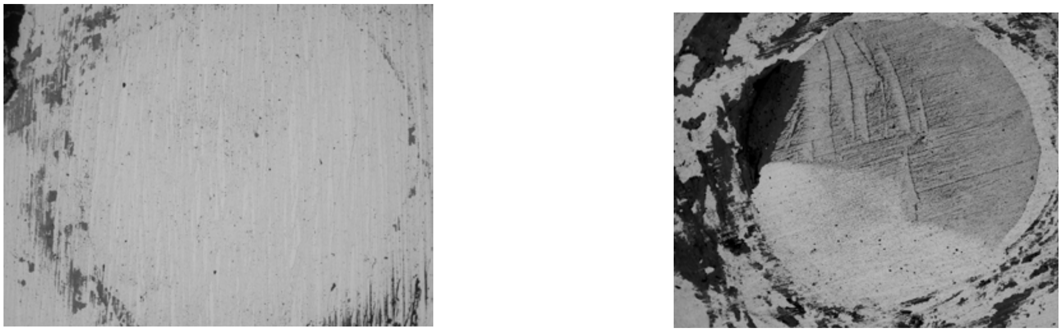

Figure 2A

Figure 2B