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The Effect of Surface Treatment of the Interfacial Surface on Fatigue-Related Microtensile Bond Strength of Milled Zirconia to Veneering Porcelain

Aaron B. Harding, DMD, MS,[1] Barry K. Norling, PhD,[2] & Erica C. Teixeira, DDS, MS, PhD[3]

[1]Assistant Professor, Uniformed Services University of the Health Sciences, United States Air Force Postgraduate Dental School, San Antonio, TX
[2]Professor, Department of Comprehensive Dentistry, University of Texas Health Science Center, San Antonio, TX
[3]Assistant Professor, Department of Comprehensive Dentistry, University of Texas Health Science Center, San Antonio, TX

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Correspondence
Aaron Harding, Dunn Dental Clinic, 1615 Truemper St, Lackland AFB, TX 78236.
E-mail: aaron.harding.dmd@gmail.com

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Abstract

Purpose: The success of zirconia-reinforced all-ceramic crowns depends on the formation of a stable bond between the zirconia core and the veneering porcelain. The purpose of this study was to test the effects of liner application and airborne particle abrasion of a postsintered Y-TZP core on the bond strength between the zirconia core and veneering porcelain with or without cyclic loading.
Materials and Methods: Kavo Everest® Y-TZP blank disks were sintered and divided into three treatment groups: airborne particle abrasion, IPS e.max® Ceram Zirliner application, or no surface treatment. The disks were then veneered with IPS e.max® ZirPress veneering porcelain. Half the veneered disks from each group were cyclically loaded. This created six experimental groups: three surface treatment groups cyclically loaded and three not loaded. The disks were then sectioned into microbars for microtensile bond strength (MTBS) testing (40 specimens per group). Specimens were luted to a fixture mount and loaded to failure using a universal testing machine (MTS Insight). The maximum force was measured and bond strength computed. Data were analyzed with a two-way ANOVA and Tukey's HSD test ($\alpha = 0.05$).
Results: Airborne particle abrasion significantly decreased MTBS values ($p = 0.043$), and ZirLiner application did not have a significant effect on MTBS values compared to control. Cyclic loading did not have a significant effect on MTBS values. The predominant failure mode in all groups was mixed.
Conclusions: Airborne particle abrasion of the interfacial surface of the Everest® Y-TZP core significantly decreased the MTBS to ZirPress veneering porcelain when compared to no interfacial surface treatment. Application of ZirLiner to the interfacial surface of the Everest® Y-TZP core did not significantly increase or decrease the MTBS to ZirPress veneering porcelain, compared to the other surface treatments. Cyclic loading did not affect bond strengths in any of the groups, regardless of surface treatment. Neither cyclic loading nor surface treatment affected the failure mode of the specimens.

All-ceramic restorations are popular, with potentially superior esthetic results and more translucency than conventional metal ceramic restorations. Since the introduction of an alumina-reinforcing phase allowed the porcelain jacket crown to become a viable restoration, many ceramic systems and manufacturing techniques have been developed.[1,2] computer-aided design/computer-aided manufacturing (CAD/CAM) restorations came in 1987 with the CEREC® system (Sirona Dental Systems, LLC, Charlotte, NC), designed to fabricate inlays. A variety of CAD/CAM systems exist on the market today, including KaVo Everest® (KaVo Dental GmbH, Biberach, Germany), introduced in 2002. These systems are used for a range of applications, including precisely milled yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) frameworks for fixed dental prostheses (FDPs).

Studies have shown that chipping of the veneering porcelain on all-ceramic restorations is more of a problem than that of porcelain-fused-to-metal restorations.[3] A chipping rate of up to 54% has been reported for Y-TZP core FDPs compared to a 2% chipping rate previously reported for metal–ceramic FDPs.[4,5]

However, a recent randomized controlled clinical trial showed the chipping rates to be more comparable: 19% for metal ceramic and 25% for Y-TZP core restorations after 40 months.[6]

The exact reason for veneer chipping seen in zirconia core restorations is unknown, but there are some core–veneer compatibility factors, which may cause chipping. Residual stresses may be an issue, because of a temperature gradient between the porcelain and the porcelain-core interface.[7] Property changes may also occur at the interface, as silica in the veneering porcelain may dissolve the stabilizing dopant (yttria) and induce a phase transformation of the zirconia or disturbing of grain boundaries, either of which could translate into chipping at the surface.[8] Chipping and delamination of the veneering porcelain from the framework are associated with a weak interfacial bond. In load-to-failure tests, catastrophic failure has been shown to occur, with cracks propagating from the outer surface toward the framework, deflecting along the core/veneer interface because of the toughness of zirconia.[9] Other laboratory and clinical studies have shown noncatastrophic failures, mostly minor surface chipping fractures, which do not reach the interface, some of which were not even noticed by the patients.[10-12] To maximize the performance of the restoration, the interfacial bond strength of the core/veneer must be adequate, especially under function.

In an effort to increase the core/veneer bond strength, different surface treatments of Y-TZP have been studied, though the effects are not yet fully understood.[13,14] The effect of airborne particle abrasion (grit blasting) on the zirconia core is disputed. It has been shown to induce monoclinic phase transformation, which may decrease the compressive strength or increase the flexural strength.[13,15-20] Grit blasting has also been shown to increase microtensile bond strength (MTBS) of untinted Y-TZP cores, but decrease the MTBS of yellow-tinted cores.[21] MTBS is a unique methodology that specifically tests for bond strength.[22]

Another investigated surface treatment is the application of a liner between the Y-TZP core and the veneering ceramic. A liner, similar in composition to veneering ceramic, is a proprietary ceramic material that some manufacturers suggest applying to the interfacial surface of zirconia to maximize bond strength, shade effects, and fluorescence. One study showed that the addition of liner decreased the bond strength to Nobel Rondo (Nobel Biocare, Yorba Linda, CA), but increased the bond strength to Cercon® Ceram (DeguDent, Hanau, Germany).[21] Regarding flexural strength, a liner has been shown to have no effect on the Lava Y-TZP (3M-ESPE, St. Paul, MN) system.[23]

The overall objective of this study is to determine the effect of different surface treatments on the interfacial bond strength between the core and veneer of an all-ceramic system, with and without cyclic loading. The primary aim of this study was to test the effects of liner application and airborne particle abrasion of a postsintered Y-TZP core on the bond strength between the core and its porcelain veneer. A secondary aim was to test whether cyclically loading the specimens before MTBS testing affects either bond strength or mode of failure (interfacial or cohesive). The null hypotheses to be tested were that there would be no significant difference in MTBS of the veneering porcelain to the zirconia core based on (1) cyclic loading, or (2) surface treatment.

Table 1 Chemical composition of materials used

Brand name	Chemical composition	Manufacturer
Everest ZS Blank	ZrO_2 (90% to 94%) Y_2O_3 (3% to 6%) Al_2O_3 (<0.5%)	KaVo
IPS e.max ZirPress	SiO_2 (57% to 62%) Al_2O_3 (12% to 16%) Na_2O (7% to 10%) K_2O (6% to 8%) CaO (2% to 4%) ZrO_2 (1.5% to 2.5%) P_2O_5 (1% to 2%) F (0.5% to 1%) Other oxides (0% to 6%) Pigments (0.2% to 0.9%)	Ivoclar Vivadent
IPS e.max Ceram ZirLiner powder	SiO_2 (50% to 60%) Al_2O_3 (16% to 22%) Na_2O (6% to 11%) K_2O (4% to 8%) CaO/P_2O_5/F (2.5% to 7.5%) ZrO_2 (1.5% to 4%) Other oxides (1.5% to 8%) Pigments (0.1% to 3%)	Ivoclar Vivadent
IPS e.max Ceram ZirLiner liquid	Water, butanediol, chloride	Ivoclar Vivadent

Materials and methods

The materials used for this investigation were a Y-TZP core, Everest® ZS, and a compatible pressable fluorapatite glass-ceramic veneering porcelain, IPS e.max® ZirPress (Ivoclar Vivadent, Amherst, NY). IPS e.max® Ceram ZirLiner was used as a surface treatment variable. ZirLiner, patented by Ivoclar in 2001 (U.S. Patent No. 6200137), is a translucent apatite glass-ceramic (CaO, P_2O_5, and F), essentially similar in composition to ZirPress, except that it is applied to the Y-TZP core as a powder–liquid paste and contains up to 4% ZrO_2 (Table 1).[24]

Three groups were prepared with different surface treatments (variable 1)—airborne particle abraded Y-TZP surface, unaltered Y-TZP surface with liner application, and no surface treatment. Each group was then subdivided into two groups: cyclically loaded (variable 2) and not cyclically loaded. This produced six total experimental groups (Fig 1).

Twelve Everest® ZS blanks (KaVo Dental) of 16 mm diameter and length were embedded in acrylic resin (Vitacrilic, Fricke Dental, Streamwood, IL) before sectioning with an Isomet 5000 saw (Buehler, Lake Bluff, IL). Six blanks were selected from each of the two manufacturer packages (Lot #101054882, 101171762). Each blank was sectioned in two, yielding 24 disks, which were then sintered according to the manufacturer's recommendations, using the Everest Therm (KaVo Dental) sintering oven. The sintered disks were randomly assigned to each of the three surface treatment groups (variable 1, 8 disks each).

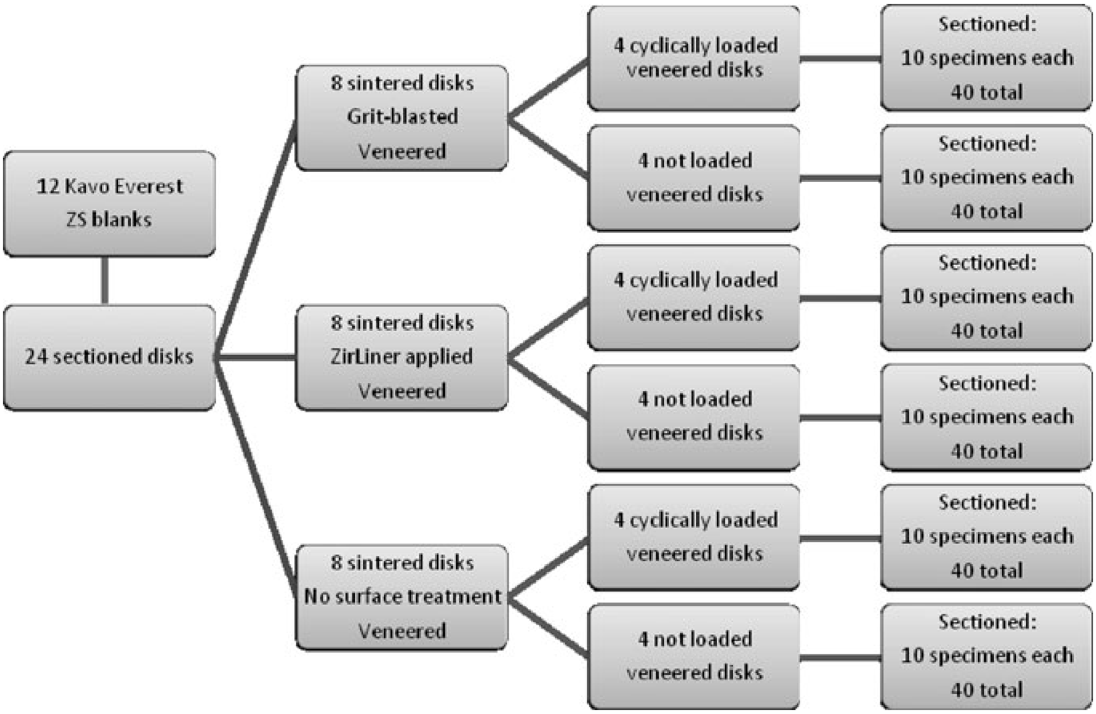

Figure 1 Experimental groups.

IPS e.max® Ceram ZirLiner was applied to the first group, and the disks were fired according to manufacturer's specifications. The surface was then sanded with 600-grit SiC paper until a ZirLiner thickness of 0.1 mm was obtained. For the second group, the entire interfacial surface of each disk was grit-blasted with 50 μm aluminum oxide particles (Lincoln Dental Supply, Myerstown, PA) at 50 psi at a standoff distance of 15 mm, for 5 seconds.[13] No surface treatment was rendered to the third (control) group.

After receiving the appropriate surface treatment, the disks were luted with sticky wax to a resin sprue pattern with a thickness of 4 mm. For standardization, a virtually designed hollow sprue pattern was generated using stereolithography. The assembly was invested in IPS® PressVEST (Ivoclar Vivadent), and placed in a burnout oven using a staged heating technique to attain a final temperature of 1562°F. The veneering porcelain (IPS e.max® ZirPress, Shade MO A2, Lot #H20589) was applied using the Programat EP 5000 furnace (Ivoclar Vivadent). The veneered disks were divested, and the sprues were removed with a diamond bur.

Each veneered disk was fully embedded in acrylic resin (Vitacrilic). Four of the eight veneered disks from each group were randomly assigned to undergo a loading cycle of 50,000 cycles at 75 cycles/min, with a spherical steel indenter under a 110 N load, in distilled water at room temperature. The veneered surface was loaded. Each load cycle included one veneered disk from each group, to minimize the effect of environmental factors such as room temperature and humidity. After cyclic loading, the veneered disks were examined for signs of obvious fractures or other defects using 20X stereoscopic magnification (Nikon SMZ-1B, Tokyo, Japan).

All eight veneered disks were then sectioned into $8 \times 1 \times 1$ mm^3 beams using a precision saw (Isomet 5000, Buehler). The specimens were examined again for defects, and unacceptable specimens were discarded. Reasons for discarding specimens were premature fracture during sectioning; chipping in the porcelain, especially notching near the interface; and porosity near the interface (Fig 2). This last phenomenon was noted particularly in the ZirLiner group.

Each disk was sectioned in such a manner as to yield a maximum of 16 specimens. Of the specimens that remained intact after preparation, ten from each veneered disk were randomly chosen for tensile loading on a universal testing machine (MTS Insight, MTS, Eden Prairie, MN). With four veneered disks in each group, this resulted in 40 specimens per group, except in both airborne particle abraded groups, which were represented by 29 specimens each, because of low specimen yield.

The cross-sectional area of each specimen was computed by recording the width of the beam at the interface to the nearest hundredth of a millimeter using a digital caliper, immediately before loading. The interfacial surface of each specimen was centered at the interface of a specialized fixture and luted to the fixture at its lateral surface near the top and bottom of the beams, using a cyanoacrylate adhesive (Zap-It, DVA, Corona, CA).[25] Each specimen was loaded to failure at a 1 mm/min crosshead speed.[21] The force at time of failure was recorded in Newtons and converted to megapascals (MPa = force/cross-sectional area). The specimens were observed under light microscopy (Nikon SMZ-1B, 20X) to determine whether failure modes were cohesive, interfacial, or mixed (Fig 3). Failure was considered to be cohesive when veneering porcelain still covered the entire interfacial surface after load to failure. Interfacial

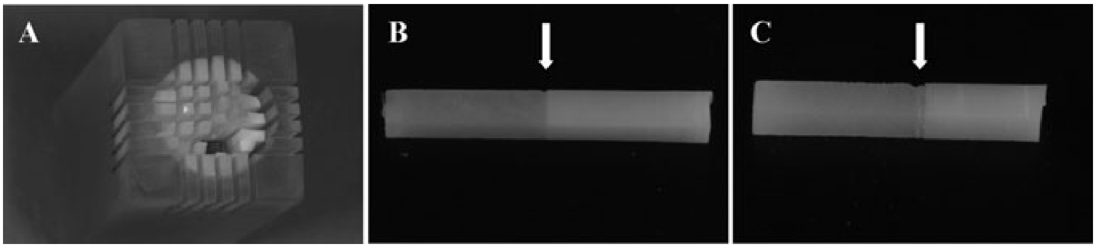

Figure 2 Discarded specimens because of (A) premature fracture, (B) notching near the interface, or (C) ZirLiner porosity.

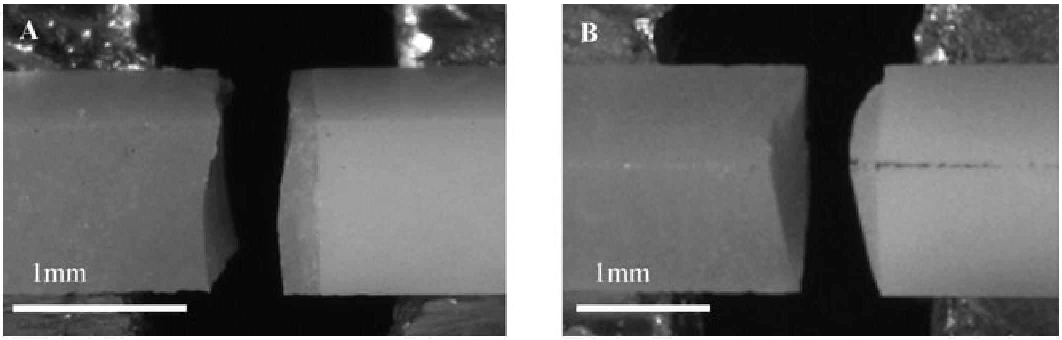

Figure 3 Microtensile bond strength testing. (A) Cohesive failure, and (B) mixed failure mode (approximately 20X magnification). No purely interfacial failures occurred.

failure was the result of a clean separation of the veneering porcelain from the zirconia. Mixed failure was a combination: some veneering porcelain remained attached to the zirconia, but some of the interfacial zirconia was visible.

Statistical methodology

The study design was a two-way factorial design. The two crossed factors were (1) surface treatment with three levels (grit-blasted, ZirLiner, and no surface treatment) and (2) pretest loading (cyclic loading vs. no cyclic loading). The data were analyzed using a two-factor ANOVA. This analysis yielded main effects tests of the surface treatment and pretest loading factors and a test of their interaction. Both omnibus ANOVA and planned pairwise comparisons of sample means were performed using Tukey's HSD test to correct for multiple comparisons. All hypothesis testing was two-sided at the 0.05 significance level.

Within each group, the percentage of specimens that failed cohesively, interfacially, or mixed was calculated. In addition, the percentage of specimens from each group that survived the specimen preparation process was reported as specimen yield.

Results

The mean $\pm$SD MTBS values are listed in Table 2. A mixed failure mode predominated in all the groups. The anticipated specimen yield was 64 for each group (4 blocks per group $\times$ 16 specimens per block). The actual yield was totaled and calculated as a percentage (Table 2). The yield was highest for no surface treatment, followed by ZirLiner and airborne particle abrasion.

There were no major outliers, and the data were not extremely skewed; however, a formal test for normality (Shapiro–Wilk test) indicated significant departure from a normal bell curve distribution. Therefore, the data were transformed by taking the square root of each value. The residuals of the transformed variable did conform to the assumptions of normality (Shapiro–Wilk $W = 0.99$, df $= 218$, $p = 0.53$) and variance homogeneity (Levene $F = 0.83$, df $= 5, 212$, $p = 0.53$).

The omnibus ANOVA yielded no statistically significant main effect of loading [$F(1, 212) = 0.59$, $p = 0.45$] or surface treatment [$F(2, 212)$, $p = 0.053$]. The third effect tested was the interaction between loading and surface treatment. This tests whether the differences among the surface treatments depend on whether the material is loaded. This test was not significant [$F(2, 218) = 0.25$, $p = 0.78$]. Thus, the first null hypothesis was accepted, and it was deemed acceptable to collapse across the loaded and not loaded samples to test for the surface treatment effect. The planned pairwise contrast showed a significant difference between airborne particle abrasion and no surface treatment ($p = 0.04$), averaging the loaded and unloaded data. Thus, the second null hypothesis was rejected. There was no significant difference between airborne particle abrasion and ZirLiner ($p = 0.49$), or between ZirLiner and no surface treatment ($p = 0.35$).

Discussion

Overall, the MTBS values (13.55 to 18.11 MPa) were on the lower end of the range previously reported in the literature for

Table 2 Number of specimens (N), MTBS values (MPa), standard deviation, specimen yield, and failure mode (cohesive, interfacial, mixed) for each experimental group

Loading	Treatment	N	Mean (MPa)	SD	Specimen yield	Failure mode C	I	M
Not loaded	ZirLiner	40	15.77	7.13	69%	3%	0%	98%
	Airborne particle abrasion	29	13.55	7.31	35%	3%	0%	97%
	No surface treatment	40	18.11	9.73	94%	5%	0%	95%
	Total	109	16.04	8.34	61%	4%	0%	96%
Cyclically loaded	ZirLiner	40	14.58	7.42	85%	0%	0%	100%
	Airborne particle abrasion	29	14.04	8.01	59%	0%	0%	100%
	No surface treatment	40	16.18	7.52	91%	8%	0%	93%
	Total	109	15.03	7.60	76%	3%	0%	97%
Total	ZirLiner	80	15.18	7.25	76%	1%	0%	99%
	Airborne particle abrasion	58	13.80	7.60	45%	2%	0%	98%
	No surface treatment	80	17.14	8.69	92%	6%	0%	94%
	Total	218	15.53	7.98	68%	3%	0%	97%

Y-TZP core–veneer specimens (16.8 to 49.8 MPa).[21] However, no known studies to date have been published that investigate the MTBS of these particular materials, so a direct comparison of the MTBS values of other zirconia and porcelain systems is difficult. In fact, a study comparing different ceramic veneering systems showed a wide range of MTBS values (14.76 to 23.52 MPa), even on a single zirconia substrate.[26] A wide variation in MTBS values appears to be the nature of this type of study when investigating Y-TZP materials. The lower MTBS values of this study also magnify the effects of standard deviation on the coefficient of variation.

The reason for the lower MTBS values is unknown, but could be a result of residual stresses introduced during specimen preparation. In general, the thicker the zirconia and veneering porcelain, the higher the residual stress.[7] It has also been shown that a higher veneer-to-core thickness ratio increases residual stresses.[27] The 5-mm thick zirconia disk and 4-mm thick veneering porcelain in this study are thicker than represented in a typical dental restoration, and would be expected to have higher residual stresses. Sectioning of the specimens involved sectioning zirconia after it had been sintered, and there may have been cracks in the zirconia core up to 15 μm, which can produce a deleterious residual stress condition.[28]

Previous studies have shown that a liner for zirconia weakens the MTBS.[26,29] The MTBS data from this study cannot statistically support either this claim or the manufacturer's claim of superior bond strength.[30] However, some of the following observations would tend to suggest a detrimental effect of ZirLiner on bond strength.

The total specimen yield for ZirLiner was 76%, which was higher than the 45% yield of the airborne particle abraded group, but lower than the 92% specimen yield of the group receiving no surface treatment. This suggests that both ZirLiner and airborne particle abrasion may interfere with the ability of the pressable ceramic to bond to the zirconia core. A recent study corroborates this finding, with decreased shear bond strength of liner application to a KaVo Everest core.[31] This finding for ZirLiner is consistent with the microscopic evaluation of the specimens before MTBS testing, some of which had porosities in the ZirLiner. These porosities may be because of the fact that ZirLiner is applied as a brushed-on paste fired like conventional porcelain. In contrast, observation of the specimens in the airborne particle abrasion and no surface treatment groups showed that the heat-pressed ceramic was much less likely to exhibit porosity near the zirconia core when there was no liner present.

The airborne particle abraded groups had a low specimen yield; only 29 for each group were able to be tested for MTBS. As the original power analysis had indicated 20 specimens per group to be sufficient to test for statistical differences, no further specimens were made. This resulted in a group size of 58 total for the airborne particle abraded group; 29 cyclically loaded and 29 not cyclically loaded. The fact that a statistical difference was found between this group and the control group is also an indication of sufficient group size. A curious finding is that the specimen yield for the airborne particle abraded group not cyclically loaded was lower than the group that was loaded. Of the four disks (not loaded) used to obtain specimens, one of them yielded no useable specimens. The reason for this is unknown, but it accounts for the lower specimen yield of the group not cyclically loaded.

The airborne particle abraded group exhibited statistically the poorest bond strength between e.max® ZirPress, and Everest® zirconia. This finding is similar to the effect on yellow-tinted Cercon® and Lava™, but the opposite of untinted Cercon®, Lava™, and Procera® cores in a previous MTBS study.[21] Although chemical characterization of the treated surface was not performed, it is possible that airborne particle abrasion could have increased the concentration of monoclinic phase zirconia, as has been shown in another study.[16] If so, transformation toughening during the sectioning process of specimen preparation would be limited. This would inhibit the ability of the zirconia to halt crack growth, and is a potential explanation for the low MTBS values.

Cyclic loading did not influence the bond strength or failure mode of the specimens. Previously published MTBS studies did not include cyclic loading of specimens under wet conditions, two factors that may enhance the clinical relevance of in vitro studies.[21,22,29,32] The loading protocol was developed based on a study that found inner cone cracks developing from the outer surface of a 0.7-mm thick porcelain veneer to its interface with a zirconia framework, without causing delamination.[33] Perhaps the 4 mm thickness of the porcelain used in this study was too great a distance for the cracks to travel to reach the interface, resulting in negligible observable effects.

Fracture analysis was readily accomplished using 20X stereoscopic magnification. No purely interfacial failures were noted. This seems inconsistent with previous studies, which noted several interfacial failures; however, those studies did not contain a "mixed" category of failure. Their interfacial failures would have been categorized as mixed in this study.[21,22,29]

Future studies should continue to focus on surface treatments that will enhance the bond strength of zirconia to veneering porcelain. It would also be worthwhile to explore whether modifications to zirconia (e.g., coloring agents) or porcelain (different shades) can negatively affect bond strength.

Conclusions

Within the parameters of this study, the following conclusions can be drawn:

(1) Airborne particle abrasion of the interfacial surface of KaVo Everest® zirconia with aluminum oxide significantly decreases the MTBS to ZirPress veneering porcelain when compared to no interfacial surface treatment.
(2) Application of ZirLiner to the interfacial surface of KaVo Everest zirconia does not significantly increase the MTBS to ZirPress veneering porcelain, compared to no interfacial surface treatment.
(3) Cyclic loading did not affect bond strengths in any of the groups, regardless of surface treatments.
(4) Neither cyclic loading nor surface treatment affected the failure mode of the specimens.

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