The increasing demands of restorations include improved esthetics and decreased biologic complications, requirements that promote the development of all-ceramic prostheses. Among the various all-ceramic materials introduced in recent years, yttrium-stabilized tetragonal zirconia (Y-TZP) has experienced substantial development based on its advantages of excellent chemical stability and high strength. Despite its desirable properties, Y-TZP has less transparency than natural teeth. Therefore, porcelain veneers are needed to obtain excellent esthetic properties. In current dental practice, zirconia restorations are fabricated using computer-aided design/computer-assisted manufacturing technologies and may be veneered with a tooth-colored porcelain for esthetic reasons. However, core-veneered zirconia restorations have the disadvantages of chipping (cohesive) and delamination (adhesive) failures of the veneer. The rates of cohesive and adhesive failures can reach 15% after 24 months, 25% after 31 months, 13% after 38 months, and 17.7% after 41 months. These failures may be caused by many factors, such as the coefficient of thermal expansion (CTE), firing process, framework surface finish and the increasing demands of restorations include improved esthetics and decreased biologic complications, requirements that promote the development of all-ceramic prostheses. Among the various all-ceramic materials introduced in recent years, yttrium-stabilized tetragonal zirconia (Y-TZP) has experienced substantial development based on its advantages of excellent chemical stability and high strength. Despite its desirable properties, Y-TZP has less transparency than natural teeth. Therefore, porcelain veneers are needed to obtain excellent esthetic properties. In current dental practice, zirconia restorations are fabricated using computer-aided design/computer-assisted manufacturing technologies and may be veneered with a tooth-colored porcelain for esthetic reasons. However, core-veneered zirconia restorations have the disadvantages of chipping (cohesive) and delamination (adhesive) failures of the veneer. The rates of cohesive and adhesive failures can reach 15% after 24 months, 25% after 31 months, 13% after 38 months, and 17.7% after 41 months. These failures may be caused by many factors, such as the coefficient of thermal expansion (CTE), firing process, framework surface finish and
pretreatments, wettability of the veneer on the core, micromechanical retention, and fatigue.13–16 Many methods have been reported to solve these problems, including airborne-particle abrasion, silica coatings, application of a liner the color of the zirconia core, and laser surface treatment.8,13,17,18 Kim et al found that the shear bond strength (SBS) between the porcelain veneer and airborne particle–blasted zirconia was significantly higher than that after application of a liner.8 However, late-generation zirconia may be susceptible to damage from grit blasting or grinding.19 Mosharraf et al suggest that the application of a liner cannot produce a significantly elevated SBS, while grinding may significantly decrease the bond strength.20 Porcelain-coated zirconia surfaces are also suspected to have improved chemical affinity.21 Therefore, the bonding between modified zirconia ceramics and porcelain veneers is still a hot topic.

The purpose of this study was to explore the effects of in situ synthesized particulates on the bonding properties between zirconia and porcelain. The null hypothesis was that the in situ synthesized particulates would have no influence on the bonding between zirconia and porcelain, the mechanical properties of zirconia, or the zirconia–porcelain interface.

MATERIALS AND METHODS

Specimen Preparation
Prefabricated Y-TZP (Nissin-Metc China), which is a highly transparent material with a CTE = 10.5 ± 0.5 × 10−6 K−1, and a porcelain veneer (Cerabien ZR, Kuraray Noritake Dental) with a CTE = 9.1 × 10−6 K−1 were used in this study. A total of 66 Y-TZP slices (10 × 10 × 1.5 mm) and 54 bar specimens for 3-point bending tests (25 × 5 × 1.5 mm) were fabricated using a diamond saw (IsoMet 4000 Linear Precision Saw, Buehler). The surfaces of the prefabricated specimens were polished with 1,200-grit silicon carbide abrasive paper (Gold Cattle, Asia Abrasive) and water cooling. The substrates were ultrasonically cleaned in deionized water and then randomly allocated into six groups (n = 11 slices and 9 bars each). The control group (C) specimens had no additional treatment after polishing. Among the experimental groups, the specimens were immersed in 40% hydrofluoric acid for 10 (I1), 30 (I3), 50 (I5), 70 (I7), or 90 (I9) seconds. Then, they were put into 0.1 mol/L calcium chloride solution for 1.5 minutes, immersed in 1 mol/L sodium hydroxide solution at 80°C in a water bath, and held for 2 hours. The specimens were then dried naturally. According to the instructions provided by the manufacturer, the samples were sintered in a sintering furnace (Everest, KaVo Dental) and ultrasonically cleaned for 10 minutes.

The 11 slices in each group underwent porcelain veneer firing. A thin layer of shade base (SB A2, Cerabien ZR, Kuraray) was applied to the modified zirconia ceramic following the instructions provided by the manufacturer. Then, a metal template (6-mm height, 12.0-mm outer diameter, and 4.5-mm inner diameter) was used to construct dentin porcelain (A3.5 B, Cerabien ZR, Kuraray) with a thickness of 4 mm. After the porcelain veneer firing process, the specimens were stored in distilled water at 37°C for 24 hours.

Surface Roughness Measurement
Before veneering, the surface roughness (Ra) of the samples was evaluated using profilometry (JB-4C, Shanghai Taiming Optical Instrument) with a 0.8-mm cut-off value and 4-mm traversing length. Each specimen was tested at three different positions, and the mean value was calculated.

Scanning Electron Microscopy
Surface Observation. Surface structures of the modified zirconia surfaces were observed with scanning electron microscopy (SEM) (Phenom ProX scanning electron microscope, Phenom-World) at ×1,000 and ×5,000 magnifications. Afterwards, 3D roughness reconstructions were photographed (Phenom ProX scanning electron microscope, Phenom-World).

Bonding Interface Observation. One of the veneered specimens in each group was selected randomly. These selected specimens were sectioned perpendicular to the sample surface, exposing the bonding interface, and wet-ground with 7,000-grit silicon carbide abrasive paper to produce a smooth surface. Micrographs of the zirconia–porcelain interfaces were photographed using SEM (at ×1,000 and ×3,000 magnifications).

Microindentation Test
After bonding interface observation, the specimens were ultrasonically cleaned, and the cross-sectional microhardness of each surface was evaluated with Vickers indentation testing (FM-700, Future-Tech) under 25 gf for 15 seconds. Ten lanes of 10 indentations each were tested, with the 10 lanes (−20, −15, −10, −5, 0, 5, 10, 15, 20, 25) at 20, 15, 10, and 5 μm into the zirconia, at the bonding interface (0), and then 5, 10, 15, 20, and 25 μm into the porcelain. A schematic diagram of the indentations is shown in Fig 1. The microhardness distributions of the interfaces were fitted with Surfer 12.2 (Golden Software).22,23

Bonding Strength Test
Shear Bond Strength. The remaining veneered specimens in the test groups were fixed and subjected to a shear bond test (n = 10). A universal testing machine (AG-Xplus, Shimadzu) was used with a 0.5-mm/minute crosshead speed until the bonding interface was
Honest Significant Difference (HSD) test was applied as a post hoc analysis. The fracture mode was evaluated with Kruskal-Wallis test, followed by Bonferroni test. Microindentation results were analyzed using one-way repeated measures ANOVA with Bonferroni test. The above statistical analyses were evaluated with SPSS 22.0 (IBM) at $\alpha = .05$.

The Weibull distribution was applied to evaluate the results of SBS and flexural strength (R 3.5.1, R Foundation for Statistical Computing) with a 63.2% probability of failure. The Weibull parameters and Weibull modulus were estimated using maximum-likelihood estimation, and 95% confidence intervals (CIs) were calculated with parametric distribution analysis (right censoring). The significant differences in the Weibull parameters and 95% CIs were compared.21

A flow chart of this study's protocol is shown in Fig 1.

RESULTS

Surface Roughness

Figure 2 shows the Ra values (mean ± SD) of all groups. The Ra significantly increased when the polycrystalline particulates were synthesized on the zirconia surface ($F = 34.133$, $P = 1.460e^{-15}$). The Ra value of group C was $0.304 \pm 0.148 \mu m$, and the values of groups I1, I3, I5, I7, and I9 were $0.388 \pm 0.136 \mu m$, $0.897 \pm 0.281 \mu m$, $1.103 \pm 0.369 \mu m$, $1.468 \pm 0.305 \mu m$, and $1.538 \pm 0.366 \mu m$, respectively. The Ra of the control group was significantly different from groups I3, I5, I7, and I9 ($P < .05$). The Ra values of groups I5 and I7 was significantly higher than that of the other groups.
the time in the hydrofluoric acid increased, the density of the particulate distribution increased.

Representative 3D images are shown in Fig 4. The surfaces in the groups were different from the reconstructed 3D images. In the experimental groups (except for I1), a uniform distribution of particles can be observed. As

SEM Observations

Figure 3 shows that different distributions of particulates were found in the experimental groups. Group I1 showed an incomplete particulate structure, while groups I3, I5, I7, and I9 offered obvious polycrystalline particulate structures. Moreover, the major axes of polycrystalline particulates in groups I5 and I7 were larger than those of the other experimental groups.

Representative 3D images are shown in Fig 4. The surfaces in the groups were different from the reconstructed 3D images. In the experimental groups (except for I1), a uniform distribution of particles can be observed. As

SEM images of the bonding interfaces are shown in Fig 5. Particulates on the surface could be observed and became integrated into zirconia substrates. As the immersion time increased, their size and crystallinity increased. However, particulates that are very crowded could easily lead to fissures, which resulted in bubbles between the zirconia substrate and porcelain (Fig 5 [I9, b1]). Bubbles could also be observed in the porcelain veneers (Fig 5 [b2]).
**Fig 4** Representative 3D images of the groups.

**Fig 5** Representative SEM micrographs of the zirconia-porcelain interfaces. P = porcelain; Z = zirconia; pp = polycrystalline particulate; b = bubble (1: between zirconia and porcelain; 2: in porcelain veneer).
**Fig 6** Hardness maps of the bonding areas (GPa).

**Fig 7** Grid microindentation results of the zirconia-porcelain interface. Transition regions of groups are marked with arrows.
acid increased was observed; however, no significant difference was found among the groups ($F = 0.064$, $P = .997$; Fig 8). The Weibull characteristic flexural strength (mean and 95% CI) values for the groups were 973.59 (914.61 to 1,032.57) (C), 971.18 (902.26 to 1,040.10) (I1), 969.81 (888.65 to 1,050.97) (I3), 952.03 (892.61 to 1,011.45) (I5), 964.69 (872.94 to 1,056.43) (I7), and 957.41 (880.27 to 1,034.55) (I9) MPa. The Weibull characteristic flexural strength and Weibull modulus values among the groups were not significantly different.

**DISCUSSION**

Ceramics, especially Y-TZP, are increasingly being applied in clinical practice as high-strength materials for dental prostheses. Porcelain veneers on a zirconia core were produced to overcome the esthetic deficiencies compared to lithia-based silicates.24–26 However, researchers have offered conflicting results on different modifications of the zirconia ceramic surface.8,13,19–21,27 Although the present authors have explored the effects of the immersion time in hydrofluoric acid and the effect of zirconia surface modifications on the bond strength and adhesive failure mode of porcelain veneers on zirconia core, the results show that the Weibull characteristic flexural strength and Weibull modulus values among the groups were not significantly different.

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of in situ synthesized polycrystalline particulates on the bonding between zirconia and resin cement, the bonding effects to porcelain were unknown. Therefore, this study was conducted to evaluate the effects of in situ synthesized polycrystalline particulates on the SBS between zirconia and porcelain and explored the interface changes.

Based on the results of this in vitro study, the null hypothesis that the method has no influence on the bonding strength of zirconia to porcelain and the zirconia-porcelain interface was rejected.

Zirconia ceramic can be etched in hydrofluoric acid without being densely sintered. The extent of the reaction in hydrofluoric acid is related to the reaction temperature, time, and concentration of the hydrofluoric acid. Therefore, zirconia ceramics in this study were treated before being densely sintered, in situ synthesized polycrystalline particulates were produced on the zirconia surface, and the reaction time in HF was explored. In the SEM images (Fig 3) and 3D images (Fig 4), regular geometric polycrystalline particulate structures were observed on the surface of the densely sintered zirconia. As the hydrofluoric acid etching time lengthened, the particulate distribution density increased as more zirconium dioxide (ZrO₂) reacted to zirconium tetrafluoride (ZrF₄). The more ZrF₄, the more ZrOCl₂ was produced after the specimens were immersed in CaCl₂ solution. Zirconium (IV) hydroxide (Zr(OH)₄) was produced by the reaction of ZrOCl₂ and sodium hydroxide (NaOH), which resulted in ZrO₂ after dense sintering. Therefore, additional polycrystalline particulates were synthesized in situ on the zirconia surface as the HF etching increased. However, the surface roughness increase was limited (Fig 2). There was no significant difference between groups I₅ and I₉. This might be due to some extent to the particulates being close to each other.

Several testing methods (such as SBS, 3-point flexural strength, 4-point flexural strength, and 2-axis flexural strength) have been used to measure core-veneer bond strength, and the values of the bond strength depend on the test method employed. This study chose the SBS method to evaluate the bond strength of different treatments. The value of the control group was similar to that reported by Aalaei et al, He et al, and Komine et al.

The bond strength increased from group C to group I₇ (Table 1). The values in groups I₅ and I₇ were higher than that of the metal-porcelain bonding in ISO 9693-1:2012. The highest mean SBS obtained herein was for group I₇ (27.02 ± 2.44 MPa), while group C showed the lowest mean SBS (20.86 ± 1.13 MPa). A previous study showed that no additional elements were found after the modification, suggesting that the surface roughness played the main role in the bond strength. In contrast, particulates on the zirconia substrates increased the bonding area and played the role of retaining nails (undercut) as well (Fig 5 [pp]), which corrected the poor bonding between coatings and substrates with traditional methods. When shear stress was applied between the zirconia and porcelain, the particulates could resist the stress, and the tops of the particulates were stretched while the bottoms were compressed. SEM images of the bonding interfaces indicated that the porcelain veneer penetrated into the zirconia substrates completely in the experimental groups (except group I₉), avoiding the possible cracks caused by voids and flaws. On the one hand, the Ra in group I₉ was slightly higher than that in I₇, but the bubbles in the bonding interface might decrease the adhesive area, resulting in poor bonding (Fig 5 [I₉, pp]). In group I₉, the particles were very close to each other, which easily caused bubbles where the porcelain veneer was attached to the zirconia substrate. The fracture behavior of the zirconia-porcelain interface was evaluated after SBS testing. Adhesive failure was the main mode for group C, and mixed failure was the main mode for the experimental groups, suggesting that in situ synthesized polycrystalline particulates on the zirconia surface could keep the porcelain connected to the zirconia well, resisting shear stresses. Observation of cohesive failure in groups I₃, I₅, I₇, and I₉ suggested that the real SBS of these groups was higher than measured, which indicated a potentially elevated bond strength in late-generation porcelains.

The greater the Weibull characteristic strength, the better the actual bonding effectiveness. An increased Weibull modulus suggests less technique sensitivity of the bonding procedure. With its elevated characteristic strength, group I₇ showed better bonding effectiveness than that of the other groups. The Weibull modulus values among the groups were not significantly different, suggesting that the technique sensitivity among groups was similar; however, the Weibull moduli of groups I₅ and I₉ were lower than that of the other experimental groups, showing a tendency of increasing sensitivity with decreased or increased immersion times in hydrofluoric acid.

The large difference in the elastic modulus between zirconia and porcelain is an important factor affecting bond stability, but modulus mapping was hard to accurately acquire. In this study, microindentation was employed to investigate the mechanical properties of the interface between the zirconia and porcelain veneer to analyze the effect of the elastic modulus on the SBS. Furthermore, the approximate mapping method was suggested by Peng as a reliable approach to gain more information on the zirconia-porcelain interface; therefore, this method was applied in this study. To avoid possible overlapping or interactions between neighboring indentations, the minimum distance between two neighboring indentations was set to 10 μm. Cross
distributions of indents were applied by moving the Berkovich diamond tip 5 μm to the right in a direction that was 60 degrees to the horizontal line (Fig 1). Finally, the space between every two columns was 5 μm, and more information was obtained. The width of the interface transition region of group C was approximately 15 μm, and the microhardness decreased rapidly from the zirconia to the porcelain. The width of the interface transition region increased with increasing hydrofluoric acid immersion time, resulting in a gradual decrease in graded microhardness. This may be due to the dense distribution of polycrystalline particulates. A gradual decrease in the graded modulus might avoid the stress concentration and protect the bonding interface; as a result, the bond strength was increased.

Interfaces with airborne-particle abrasion could decrease fracture toughness due to the existence of flaws and voids.35 Wang et al claimed that the size and shape of flaws and voids influence the formation of cracks.35 There were no obvious voids obtained in the experimental groups. However, flaws could be found in group Igr, which resulted in a decreased SBS. Moreover, the particulates protruded on the surface of the matrix and could become stress concentration sources due to the large difference between zirconia and porcelain.

The flexural strengths were slightly lower in the experimental groups but were not significantly different, thus confirming the null hypothesis that in situ synthesized polycrystalline particulates affect the mechanical properties of zirconia. The thickness of the specimen decreased after hydrofluoric acid etching, resulting in a slight decrease in the mechanical properties. Another reason might be that the immersion treatment changed the structure of the presintered zirconia. Weibull analysis showed that the flexural strengths of the experimental groups were at the same level.

CONCLUSIONS

Within the limitations of this study, in situ synthesized polycrystalline particulates on zirconia ceramic acted as retaining nails, increased the Ra and width of the interface transition region, and prevented a sharp decrease in the elastic modulus around the bonding interface. These findings suggest potential in dental applications. Additionally, the approximate mapping method of indentations is a practical way to explore the interface between zirconia and porcelain veneers. Further studies are still needed to evaluate the impact of the porcelain variables of low-temperature aging, thermocycling, and stress fatigue.

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REFERENCES


Literature Abstract

Effect of Previous Implant Failure on the Prognosis of Subsequent Implants: A Retrospective Study

The aim of this study was to compare the long-term survival of dental implants placed in patients with and without a history of implant failure. Within a retrospective analysis, an experimental group was selected consisting of 59 patients with 137 implants placed after previous failures. The control group included 1,072 patients with 2,664 implants without previous failure. Kaplan-Meier curves were used to describe the group-specific, long-term implant survival. Mixed-effects Cox regression models were applied to examine the effects of patient- and site-specific risk factors. To take multiple implants into account, a random intercept model was applied. During the observation period of up to 15 years, 11 implants (8%) failed in the experimental group and 74 implants (2.5%) in the control group (P < .001). The five-year cumulative survival was 96.8% (95% CI: 0.96–0.98) in the control group and 91.5% (95% CI: 0.86–0.97) in the experimental group. The variables group assignment and simultaneous augmentation had a significant effect on survival, but this effect was lost in the random intercept model. The effect of implant location remained, whereby the risk of failure was five times lower for mandibular implants, irrespective of group (P = .013; 95% CI: 0.103–0.767; HR: 0.281). Conclusions: Long-term implant survival was lower in the experimental group than in the control group. The effect of previous failure was negligible. However, a patient-specific “clustering effect” was observed. Irrespective of previous implant failures, the risk of long-term failure is two times higher for maxillary implants than for mandibular implants.


Literature Abstract

Hygiene Recall in Diabetic and Nondiabetic Patients: A Periodic Prognostic Factor in the Protection Against Peri-Implantitis?

Diabetes mellitus is associated with an increased risk of poor outcomes with dental implant placement. This study aimed to identify whether frequency of hygiene visits is a protective factor for the development of peri-implantitis in patients with diabetes mellitus. A retrospective cohort design was conducted in patients presenting for dental implant placement at the Philadelphia Veterans Affairs Medical Center from 2006 to 2012. The primary predictor variable was hygiene frequency, recorded as either infrequent, annual (7 to 12-month recall), or biannual (≤ 6-month recall). The number of months between implant placement and the presence of peri-implantitis was the primary outcome variable (time to peri-implantitis), which was assessed on a subject level and adjusted for clustered, correlated multiple implants in the same subject. Additional variables were greater ≥ 60 years of age, male gender, smokers, short implant length, diabetes mellitus, uncontrolled diabetes mellitus, and removable prostheses. Descriptive, univariate, and Cox proportional hazards regression statistics were computed to measure associations with peri-implantitis, with P ≤ .05 used to define statistical significance. The study sample was composed of 286 patients. In total, 748 implants were placed. Subjects ≥ 60 years of age were 2 times more likely to develop peri-implantitis (hazard ratio [HR] = 2.015, 95% CI = 0.985–4.119, P = .0549). Subjects receiving implant-supported removable prostheses were 2.3 times more likely to develop peri-implantitis (HR = 2.315, 95% CI = 1.006–5.327, P = .0485). With each hygiene visit, patients’ risk of developing peri-implantitis decreased by 20% (HR = 0.805, 95% CI = 0.394–1.647, P = .5528). In addition, patients with diabetes mellitus were 49% more likely to develop peri-implantitis (HR = 1.491, 95% CI = 0.758–2.936, P = .2475) than nondiabetic patients. Diabetic patients may be at increased risk for the development of peri-implantitis, and an increased frequency of hygiene visits may reduce peri-implant disease.