Effects of a convenient silica-coating treatment on shear bond strengths of porcelain veneers on zirconia-based ceramics

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Porcelain veneering of zirconia is necessary, but chipping of the veneer may cause clinical problems. We investigated the effects of silica coating to improve the bond strength between porcelain veneers and zirconia-based ceramics. The ceramics tested were zirconia/alumina nanocomposites stabilized with ceria. Three surface treatments, grinding with a carborundum point (CA), sandblasting with alumina (SB), and Silano-Pen treatment (SP), were performed. Untreated specimens (NT) were examined as a control. The surface roughnesses and contact angles after treatment were measured. Shear bond tests were conducted, and the average strengths were calculated. EPMA was used for elemental identifications and surface observations. The bond strengths with SP were 20.00 ± 3.43 MPa and were significantly larger than that (15.35 ± 3.12 MPa) of NT ($p < 0.05$). However, differences among treated specimens were not significant. Moreover, SP gave superior wettability, and smoothness as good as NT. These data suggest that SP affects the bond strength between zirconia and a porcelain veneer.

Keywords: Zirconia-based ceramics, Shear bond strength, Surface texture, Silica coating, Porcelain veneer

INTRODUCTION

Significant progress has been achieved in ceramics since the late 1990s¹⁻⁴. In particular, new technological innovations such as heat-pressed^{5,6}, injection-molded⁷, slip-cast⁸, and glass-infiltrated ceramics⁹⁻¹¹ have widened the use of ceramics in prosthetic and operative dentistry. However, properties such as brittleness limit their use in applications such as fixed partial dentures.

Since the beginning of the 21st century, zirconium dioxide (ZrO_2 : zirconia), a new and tough ceramic material, has been receiving increasing attention in dentistry¹²⁻¹⁶. Initially, its use was restricted to metal-free fixed partial anterior dentures, but its use is being extended to a variety of applications, such as fixed partial posterior dentures, orthodontic brackets¹⁹ and dental implant abutment or fixture^{20,21}, as well as simple dental restoration or prostheses^{17,18}. Above all, frameworks using zirconia-based ceramics have gained popularity for use as metal-free, long-lasting, and extensive fixed dental prostheses.

Zirconia-based ceramics are characterized by a tetragonal phase, a monoclinic phase, and a cubic phase, and these phases are mutually interchangeable under some kinds of stress such as heating, sandblasting, and mechanical grinding. It is also well known that volume changes occur when going from monoclinic to tetragonal crystals under the specific actions mentioned above^{22,23}. This phase change is useful for controlling crack propagation in zirconia-based ceramics and contributes to their high fracture toughness. Formation of the tetragonal phase strongly stabilizes zirconia-based ceramics, but other crystal phases are undesirable. To

maintain the tetragonal phase at room temperature, zirconia-based ceramics must be stabilized by adding oxides. Yttria is a popular stabilizing additive, giving Y-TZP^{12,13,16}. This new ceramic is useful as a prosthetic frame material because of its high fracture toughness and its precision fabrication with dental CAD/CAM systems. A final prosthetic process is needed to deposit a porcelain veneer on zirconia-based ceramics; however, chipping of the porcelain veneer causes clinical problems. Hydrothermal degradation of the veneer with low-temperature aging is also controversial^{16,24}. To overcome these problems, some modification techniques have been proposed.

The latest zirconia-based ceramics materials, stabilized by ceria, were developed to achieve better mechanical properties than those of Y-TZP²⁵⁻²⁸. Ceria-stabilized tetragonal zirconia/alumina nanocomposites (zirconia nanocomposites) are significantly reinforced by dispersing different nanosized particles into each crystal grain. Furthermore, preparation, prior to veneering, of frameworks as thin as those of traditional porcelain-fused metal (PFM) is possible because of the flexibility of these nanocomposites, so extremely light frames of thickness 0.3 mm can be obtained.

In esthetic restoration, dental PFM has served the profession well for nearly a half-century, but there are many disadvantages to using metals, such as allergic reactions as a result of the release of harmful ions into the body and/or low durability. Most dentists prefer all-ceramic restoration to traditional PFM methods when attempting to achieve the optimum esthetic results. Generally speaking, fixed-type all-ceramic prostheses usually give better esthetic results than PFM because of the difficulty in blocking out the color and opacity of the metal substructure. Moreover, it has been reported that

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the marginal regions of ceramics are compatible with gingival tissues²⁹.

Porcelain veneering of zirconia-based ceramics is necessary because of their opaque white color and hardness. Much research has already been done on porcelain fused to zirconia^{30–35}. Various surface modification techniques have been recommended, such as sandblasting with different particle sizes and different oxides^{30,31,34,35}, mechanical grinding³⁵, application of hydrogen fluoride³⁴, and heat-treatment³⁰. One report suggested that coarse surfaces contribute to increased bond strength³⁵, but other reports asserted that no significant effect was found^{30,31}. In the case of zirconia-based ceramics, there have been few reports on the effects of surface modification. Most techniques can certainly damage the outer surfaces of zirconia-based ceramics^{30,31,34,35}, and may spoil extremely thin frames. Consequently, an additional surface modification, such as a coating, is expected to be reasonable and to effectively increase the bond strengths between zirconia-based ceramics and porcelain veneers.

Recently, a convenient silica-coating procedure, using a silane-coupling agent, has been developed in Europe and used to improve the bond strength between a metal and a resin composite for crowns^{36–38}. This method involves kinetic spraying, and surface modification is carried out by thermal spraying with liquefied petroleum gas containing a fine silica powder. No disadvantageous morphological changes occur during the preparation because the silicate layer is formed on the surface using a flame. We assume that this method is appropriate

for joining dental porcelain to zirconia-based ceramics because silicon is the key element in dental porcelain. The purpose of this study is to investigate the effects of a silica-coating layer for the bond strength between porcelain veneers and zirconia-based ceramics.

MATERIALS AND METHODS

Specimen preparation

A ceria-stabilized tetragonal zirconia/alumina nanocomposite (P-NANO ZR (NZR), Panasonic Healthcare, Osaka, Japan) and a dental porcelain veneer (Cerabien ZR (CZR), Noritake Dental Supply Co., Ltd., Nagoya, Japan) for zirconia-based ceramics were selected for this study. The zirconia-based ceramics and veneering porcelain tested are listed in Table 1. Forty zirconia-based ceramic specimens of dimensions 11×11×2 mm³ were separated using a grinding machine (F-524HDP, Nicco Co., Ltd., Niigata, Japan) with an exclusive cutter (SDC170, Nicco Co., Ltd., Niigata, Japan) from full-sintered zirconia bar (diameter: 12 mm, length: 76 mm) for the shear bond tests. Each specimen was finally polished by another grinding device (F-515HDIP, Nicco Co., Ltd., Niigata, Japan) using two kinds of whetstone (#325, #1,000). These specimens were classified into four groups, according to the different surface treatments.

Details of the surface treatments are as follows. The CA specimens were zirconia-based ceramic specimens ground vertically and horizontally for 10 s with a carborundum point (HARD HP 13, Shofu Inc., Kyoto, Japan) using a commercial rotation device (Lab Master,

Table 1 Materials used in this study

Material	Brand	Lot number	Manufacturer
ZrO ₂	P-NANO ZR (NZR)		Panasonic Healthcare Co., Ltd.
Porcelain	Cerabien ZR (CZR)		
	Powder: A2B	16866	Noritake Dental Supply Co., Ltd.
	SB A3	19808	
	Liquid: Forming liquid	BRMVN

Fig. 1 Schema of Silano-Pen treatment tested in this study.

Yoshida Co., Ltd., Tokyo, Japan). The SB specimens were sandblasted for 10 s at an air pressure of 0.2 MPa with alumina particles of average particle size 50 μm , using a commercial sandblasting apparatus. The distance between the nozzle and the surface was set at 10 mm. The SP specimens were coated for 10 s using a frame-coating device (Silano-Pen, Bredent GmbH & Co. KG, Senden, Germany); the Silano-Pen and coating mechanism are shown in Fig. 1. The principle is approximately equal with previous Silicoater treatment³⁹. A silicate layer is quickly formed by spraying with fused silicon dioxide. Untreated specimens, coded NT, were used as a control. All the specimens modified using each treatment was ultrasonically rinsed by an ultrasonic cleaner (US cleaner USD 1R, AsOne Co., Ltd, Osaka, Japan) with acetone for 3 min, except in the case of SP processing.

Surface roughness

The surface roughness values (R_a) of the zirconia-based ceramic specimens for each modified surface were evaluated using a surface-texture and contour-measuring instrument (Surfcom 480A, Tokyo Seimitsu Co., Ltd., Tokyo, Japan). The cut-off value for the surface roughness was set at 0.8 mm, and the traversing length of the stylus with tip radius of 2 μm was 4 mm. The measurements were performed at five different points, and the average values were calculated.

Contact angle measurements

Prior to the process of building up porcelain on the zirconia-based ceramics, the wettability of each modified surface was investigated by measuring the contact angle using a contact-angle meter (SI Image Mini, Excimer Inc., Yokohama, Japan). Deionized water (5 μL) was dropped onto the center of the specimen using an attached injector. In consideration of energy change with time dependency, each image was taken and recorded 10 s after injection of a drop onto the specimen. The contact angles were gauged from the captured images using goniometry software, and were provided as average values with their standard deviations. The measurements were repeated three times for each specimen.

Surface topography analyses

To evaluate the surface topography, specimens modified using each treatment were sputter-coated with gold using an ion-sputtering device (Ion Coater, Eikoseiki Co., Ltd., Tokyo, Japan).

After completing the surface treatments and shear bond tests, elemental analyses for zirconium, aluminum, and yttrium were performed using an electron-probe microanalyzer (EPMA1610, Shimadzu Co., Ltd., Kyoto, Japan). Silicon was also determined for the SP-treated specimens. The experimental conditions were as follows: voltage 20 kV, specimen current 50 nA, scanning step interval 1 μm . Simultaneously, the surface texture of the area being analyzed was observed to check the morphology of the specimen from secondary electron (SE) images.

Shear bond tests

To evaluate the shear bond strengths, a metal cylinder (6 mm height, 12.0 mm diameter) with a hollow (6 mm diameter) was centered on the each specimen. The shade base powder of CZR was kneaded with attached liner liquid and built up to a thickness of 1 mm by using an ultrasonic condenser (CeraconII, Shofu Inc., Kyoto, Japan) on zirconia-based specimens modified by each of the treatments mentioned above, and fired in an automatic furnace (KDF Master Spirit, Yoshida Co., Ltd., Tokyo, Japan). The height of initial layer after firing was adjusted to 0.5 mm by grinding with rotary tools. A thickness of ceramic layer was checked by a caliper (ABS Digimatic Caliper, Mitutoyo Corp., Kawasaki, Japan). A second layer, up to a thickness of 5 mm, was similarly constructed. The heating conditions are shown in Table 2. Each specimen was embedded in an acrylic tube mold using methyl methacrylate resin (Unifast Trad, GC Co., Ltd., Tokyo, Japan) and subjected to bonding tests. The detailed procedure is shown in Fig. 2.

Shear bond tests were performed using a universal testing machine (Instron MD1125, Instron Japan, Kawasaki, Japan) with a 2-kN load cell at a crosshead speed of 1 mm/min, until separation of the veneer from the zirconia-based ceramic occurred. A force was applied to the specimen so that the shear load was exerted adjacent to and directly to the bonding interface. The

Table 2 Surface treatments for CZR

Code	Each procedure
CA	Mechanical grinding with a carborundum point for 10 s
SB	Sandblasting with alumina particle (50 μm) for 10 s
SP	Flame-coating with silane for 10 s
NT	Non treatment

Table 3 Heating condition for CZR

Heating schedule	Shade Base	Dentin
Standby temp. (Vacuum strating)	600(°C)	600(°C)
Drying time	5(min)	7–10(min)
Heating rate	45(°C/min)	45(°C/min)
Maximum firing temp.	930(°C)	930–940(°C)
Holding time	1(min)	1(min)
Vacuum cancelling temp.	930(°C)	930–940(°C)

Fig. 2 Specimen fabrication for shear bond tests.

Fig. 3 Surface roughness after each surface treatment. Vertical bars indicate the standard deviation and identical letters indicate that the values are not significantly different ($p>0.05$). Fig. 4 Contact angle after each surface treatment. Vertical bars indicate the standard deviation and identical letters indicate that the values are not significantly different ($p>0.05$).

Fig. 5 Typical images of wettability for each treatment.

shear bond strength was calculated as an average value from 10 data.

Statistical analyses

Statistical assessments were carried out using one-way analysis of variance. Subsequently, Tukey's HSD multiple comparison was applied to each of the parameters to compare the different surface treatments. Each test was estimated at a level of significance of 0.05.

RESULTS

A surface-roughness comparison is shown in Fig. 3.

The data were clearly divided into two significantly different types. One is an uneven surface, produced by SB and CA, and these R_a values were $0.46 \pm 0.06 \mu\text{m}$ and $0.41 \pm 0.07 \mu\text{m}$, respectively. In contrast, the value for SP was $0.11 \pm 0.02 \mu\text{m}$ and then was close to that for NT, which was $0.09 \pm 0.02 \mu\text{m}$; the difference between them was not significant ($p > 0.05$).

The contact-angle measurements are shown in Figs. 4 and 5. A noteworthy property is the excellent wettability of the SP specimen, which could not be measured and was far superior to those of the other specimens. The contact angle of the CA and NT specimens were the same as each other, and were larger than that of the SB

Fig. 6 (a) Low-magnification ($\times 500$) SE images after each surface treatment.
(b) High-magnification ($\times 5000$) SE images after each surface treatment.

Fig. 7 High-magnification SE images.

Fig. 8 Shear bond strength for each specimen. Vertical bars indicate the standard deviation and identical letters indicate that the values are not significantly different ($p > 0.05$).

specimen.

Representative SE images of each specimen are shown in Figs. 6a and 6b. In the low-magnification photographs, there is no apparent difference between SP and NT; however, specific differences between their surface qualities were observed in high-magnification photographs. The SB and CA specimens had distinctive and uneven surface scraped by abrasive technology. They were observed with the low magnification clearly.

Elemental distribution results for each group are presented in Table 3. Basically, there were no large discrepancies among NT, CA, SB, and SP. According to former reports²⁵⁻²⁸, ceria-stabilized tetragonal zirconia/alumina nanocomposites consist of three elements, namely zirconium (Zr), aluminum (Al), and yttrium (Y). Silicon (Si) was not detected in specimens which did not undergo SP treatment. High magnification SE images of specimens after SP treatment are shown in Fig. 7. The images indicate that many nanosized fine grains were scattered and piled up, forming a stratified structure.

Figure 8 shows the results of shear bond strength tests. The highest strength was 23.14 ± 5.39 MPa, obtained for CA, and those of the others decreased in the order SB (20.46 ± 2.79 MPa) > SP (20.00 ± 3.43 MPa) > NT (15.35 ± 3.12 MPa). The value for NT was significantly lower ($p < 0.05$) than those for the other groups; CA ($p = 0.001$), SB ($p = 0.017$), and SP ($p = 0.041$). In addition, there were no significant differences among treatment groups.

SE images of the specimens after the shear bond tests are shown in Fig. 9. The surfaces of the SB, CA, and SP specimens are completely covered by veneering materials. Based on the images shown in Fig. 6a, it was surmised that this surface-covering layer would be a residual substance from the porcelain veneer. In contrast, some scratches seen on the original surface shown in NT of Fig. 6b were clearly exposed in the NT specimen of Fig. 9.

Figure 10 compares NT with SP using mapping analysis for Zr, Al, and Si. The failure type for NT should be mixed fracture because Zr is predominant. On the other hand, it was concluded that the SP treatment led to cohesive failure since little Zr was detected on the surface.

DISCUSSION

Methods such as shear bond tests³⁰⁻³⁵, bending tests³⁸, and tensile tests with push-out tests³⁹ are used to estimate bond strength. In this study, shear bond tests were chosen due to not only many reports³⁰⁻³⁵ in the same experimental category but severe testing method. The specimens after grinding or sandblasting had large surface roughness values and shear bond strengths compared with those of the control, and had large. Fischer *et al.* reported³¹ that the bonding strength of dental porcelain to zirconia-based ceramics did not depend on the surface roughness, but was affected by the application of opacity³¹. In contrast, our data suggested that a high surface roughness successfully improved the bonding strength. Shear bond strength values obtained in this study do not have a major difference in comparison with other similar reports^{30,31}, and thus the validity of our experiments has been proven indirectly.

Fig. 9 SE images of specimen surfaces after shear bond tests.

Fig. 10 Element mapping for NT and SP surfaces after shear bond tests.

The shear bond strength is also improved by SP processing, although the average surface roughness value of SP treatment was $0.11 \pm 0.01 \mu\text{m}$ and almost equal to that of the control. This suggests that the surface roughness of zirconia-based ceramics before fusing to porcelain may not be of great significance. Therefore, it was also found that surface coarseness of zirconia-based ceramics may not be a factor of the shear strength improvement. The contact angles were then measured to explore the superiority of SP processing.

When the surface energy of the parent material is

large, the wettability is excellent. Because the SP process directly fixes a silane layer composed of fine silicon grains by flaming with gas, it is expected that the target surface will be temporarily activated. In fact, the existence of innumerable nanosized protrusions was clearly observed in the SE images in Fig. 7. The contact angle therefore was small, and favored extension with dental porcelain powder mixed with distilled water. The contact angle on the SP processed surface was undetectable. Chemical coupling to the dental porcelain is also expected because of silica layer formed on the zirconia-based ceramics

treated by SP, as shown in Fig. 10 and an electron probe microanalyzer of Table 3. The zirconia-based ceramics modified by SP therefore had a more compatible surface than those produced by the other procedures, and the surface was considered to be advantageous for fusing to porcelain because it was unnecessary for a thin frame to be exposed to mechanical forces such as blasting or grinding, intentionally.

This prediction is supported by the results of surface observation performed after the shear bond tests, shown in Fig. 10. Cutting lines traced when making the specimens were clearly observed on the NP surface after shear bond examination; however, it was not found in other specimens. This proves that there is partial adhesive failure in the NT specimen. In contrast, the specimens treated by SP processing were almost as smooth as the NT specimens, and were entirely covered by the porcelain veneer after the bonding tests. It was therefore considered that the SP treatment conducted cohesive failure. These results suggest that SP treatment is simple and safe enough for use on zirconia-based ceramics to be fused with dental porcelain, since SP treatment gives excellent wettability and new layer formation coated by silica on the surface.

In this study, preparation of the specimens for the shear bond tests started about 1 h after rinsing in acetone solution was performed. Additionally, the ultrasonic rinsing process was omitted in the SP treatment because it was still uncertain whether the silica layer produced by SP strongly adhered to the surface. Moreover, it was thought that the surface energy would decrease with time, and treatment effects would fade. Detailed mechanism for increasing the bond strength between veneering porcelain and NZR is also indefinite. Furthermore, fatigue test with repetitive loading and the heat stress like thermocycling that simulated a clinic situation are not carried out. Consequently, we will be performing further examinations in the near future to clarify these points.

Within this limited study, we concluded that SP treatment was possible to increase the bond strength between veneering porcelain and zirconia-based ceramics without damaging zirconia surface.

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