

Improvement of Bond Strength between Zirconia Framework and Veneering Porcelain

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Abstract

A series of specimens with different surface morphology, pre-sintering process, and sintering temperature were designed for studying the improvement of bond strength between zirconia framework and veneering porcelain. Shear strength test, Scanning Electron Microscope (SEM), and Energy Dispersive Spectrometer (EDS) were adopted in the investigation. A remarkably enhance of average bond strength from 24.1 MPa to 37.2 MPa was obtained by performing humps on zirconia substrate. Moreover, a pre-sintering process for bonding porcelain was beneficial to the increasing of bond strength. Hence, during all-ceramic dental restoration fabrication, it is promising to perform surface treatment on zirconia and conduct pre-sintering procedure on bonding porcelain for the improvement of bond strength between zirconia framework and veneering porcelain.

Keywords: Zirconia restoration; Veneering porcelain; Bond strength

Introduction

Clinical statistics show that, the defect and absence of tooth is a common disease with a high morbidity of 24-53% in dental treatment. Thus, it is promising to develop dental restoration materials with favorable mechanical strength and excellent bio-safety [1-4]. In the past decades, metals and alloys have been widely used in dental restorations. However, with the increasing esthetic requirement from patients and the development of CAD/CAM (Computer Aided Design/Computer Aided Manufacturing) technique, various all-ceramic materials have been adopted in dental restoration for their aesthetical potential [5-7]. Among numerous dental ceramics, zirconia based materials possess most promising prospects due to their fascinating aesthetic performance, favorable biocompatibility, and superior mechanical property [8].

Commercially used dental zirconia usually contains 3-6 mol% of Ytria as the stabilizer, since Ytria partially stabilized tetragonal zirconia polycrystalline (Y-TZP) showed better mechanical properties and fracture toughness compared to other dental ceramics [9]. The excellent mechanical strength of Y-TZP originated from the crystalline phase transformation from a tetragonal to a monoclinic structure. Due to unsatisfying translucency of Y-TZP, a bilayered structure consisting of a zirconia substrate and a brittle glass-ceramic veneering layer is frequently used to improve the aesthetical appearance of zirconia prosthesis.

In vitro investigations indicated that Y-TZP holds flexural strength ranging from 900-1200 MPa and fracture toughness of 5-10 MPa/m². However, as reported by Sailer et al. the failure incidence for zirconia ceramic restoration in 3 and 5 years turned out to be 13.0% and 15.2%, respectively [10]. Short-term and mid-term clinical observations indicate that chipping and fragmentation of veneering porcelain were the most common reasons for the failure of all-ceramic restorations. To decreasing the risk of chipping, it is crucial to improve the bond strength between zirconia and veneering porcelain [11-13].

In this study, a series of specimens with zirconia substrate and veneering porcelain were prepared. Different surface morphology, pre-sintering process, and sintering temperature were designed. The shear strength test indicated that, the striations made no obvious difference to bond strength, while humps on zirconia substrate could obviously

enhanced the bond strength between zirconia substrate and veneering porcelain. Due to the element diffusion, which was confirmed by Energy Dispersive Spectrometer (EDS), the bond strength was increased by a pre-sintering process for bonding porcelain. Moreover, the sintering temperature of veneering porcelain was optimized to be 960°C.

Materials and Methods

Specimen preparation

Partially-sintered Kavo zirconia blocks (Kavo Corp., Germany) with the same batch and the same after-sintered size of 11×10×5 mm were used in this study. The specimens were cut and milled by Kavo CAD/CAM system (Kavo Corp., Germany). Afterwards, the specimens were ground by 120, 200, 400, 600, 800, and 1000 grit SiC paper sequentially, and polished adopting 15 μm diamond paste. They were sandblasted by alumina particles of 120 μm at 0.15 MPa for 20 seconds. Finally, the specimens were cleaned by ultrasonic cleaning for 10 min, and dried by infrared drying light. The veneering process was performed using KISS veneering porcelain (Degudent Corp., Germany) on the fully sintered zirconia, and the sintering process was performed by Ney Centurion Q 50 porcelain furnace (Ney Dental Inc, American).

To investigate the effect of surface morphology on the bond strength, 40 specimens were divided into 4 groups (Group 1.1, Group 1.2, Group 1.3, and Group 1.4). Group 1.1 was designed with striations (width of striations: 8 μm, space between striations: 160 μm) vertical to

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the orientation of shear force. Group 1.2 was designed with striations (width of striations: 8 μm, space between striations: 160 μm) parallel to the orientation of shear force. Group 1.3 was designed with cylindrical humps (diameter: 2 mm, height: 0.3 mm). Group 1.4 with no surface treatment was adopted as control group. After surface treatments by scheduled procedure of CAD/CAM system, the specimens were coated with bonding porcelain, and sintered at 980°C for 1 min, and then, the veneering layer was built in the size of 10×5×4 mm near the bonding porcelain, and sintered at 980°C for 1 min.

To investigate the effect of pre-sintering process for bonding porcelain on the bond strength, 20 specimens were divided into 2 groups (Group 2.1 and Group 2.2). Both groups were cut, ground, polished, sandblasted and cleaned by the same way mentioned above. Group 2.1 was adopted as the control group. After cleaning and drying, specimens in Group 2.1 were pre-sintered at 1000°C for 15 min directly, while specimens in Group 2.2 were coated with bonding porcelain, and then pre-sintered at 1000°C for 15 min. After cooling into room temperature, the specimens both in Group 2.1 and Group 2.2 were covered by veneering porcelain (10×5×4 mm) and sintered at 980°C for 1 min.

To investigate the effect of sintering temperature on the bond strength, 110 specimens were divided into 11 groups (Group 3.1 to Group 3.11). After the general processing mentioned above, the specimens in 11 groups were coated with bonding porcelain, and pre-sintered at 1000°C for 15 min. After cooling into room temperature, the specimens from Group 3.1 to Group 3.11 were covered by veneering porcelain (10×5×4 mm) and sintered at the temperature of 900°C, 910°C, 920°C, 930°C, 940°C, 950°C, 960°C, 970°C, 980°C, 990°C and 1000°C, respectively, for 1 min.

Testing method

The fracture surface appearance observation and energy spectrum analysis of specimen was carried out by JSM-6310F SEM.

To perform the shear strength test, the specimens were put into the fixture, and the fixture was laid on the plinth of the universal testing machine. The load was vertical to the bonding interface at the speed of 0.1 mm/min referring to criterion of YY0716-2009. The maximum shear loads were taken notes when the samples were broken down. Shear strength was obtained by the formula of $P=F/S$ (MPa), where P is the shear strength, F is the ultimate shear load, and S is the area of veneering porcelain.

SPSS 13.0 software was used for statistical analyses, and least significant difference (LSD) test between groups were performed to compare the shear strength of specimens in different groups. Pearson correlation was introduced in this study, and the $p>0.05$ indicating no significant difference between data, and there was no statistical significance, while the $p<0.05$ indicating significant difference between data, and there was statistical significance.

Results and Discussion

Effect of surface morphology on the bond strength

Figure 1 shows the surface treatments of zirconia substrates in Group 1.1, 1.2, and 1.3. As shown in Table 1, the average values and standard deviations of shear strength for specimens in Group 1.1 to 1.4 were measured to be 27.9 ± 4.53 MPa, 27.5 ± 4.79 MPa, 37.2 ± 7.03 MPa and 24.1 ± 2.64 MPa, respectively. The average shear strength of Group 1.3 was obviously higher than those of Groups 1.1, 1.2 and 1.4. The p-value between each two groups from 1.1 to 1.4 was shown in Table

2. Obviously, there is statistical significance of shear strength between Group 1.3 and Group 1.1, 1.2, and 1.4, respectively. It is safe to say that, the striations made no obvious difference to the bond strength, while humps in the surface of zirconia substrate remarkably enhanced the bond strength between zirconia substrate and veneering porcelain. Therefore, it is promising to design humps on zirconia substrate to reduce chipping of veneering porcelain.

Figure 2 showed the appearance of fracture surface after shear test. Residual veneering porcelains were found in the surface of zirconia frameworks, the residual veneering porcelain on zirconia framework in Group 3 was especially more.

Effect of pre-sintering process on the bond strength

The average values of shear strength for specimens in Group 2.1 and 2.2 were measured to be 24.78 ± 3.60 MPa and 43.12 ± 4.50 MPa,

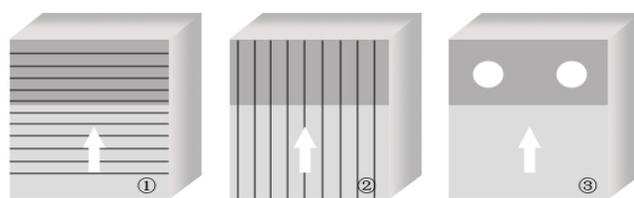


Figure 1: Surface morphology of zirconia bodies in Group 1, 2, and 3 after treatments.

Group No.	Maximum (MPa)	Minimum (MPa)	Average (MPa)	Standard deviation
1.1	35	23	27.90	4.53
1.2	36	23	27.50	4.79
1.3	49	30	37.20	7.03
1.4	28	21	24.10	2.64
Value of F				12.613*

*: significant difference ($p<0.05$)

Table 1: The average values and standard deviations of shear strength for specimens in Groups 1.1 to 1.4.

Group No.	1.1	1.2	1.3	1.4
1.1	-	0.18	0.008	0.32
1.2	ns	-	0.009	0.44
1.3	**	**	-	0.42
1.4	ns	ns	*	-

ns: no significant difference ($p>0.05$); *: significant difference ($p<0.05$); **: very significant difference ($p<0.01$).

Table 2: p-value between each two groups from Groups 1.1 to 1.4.

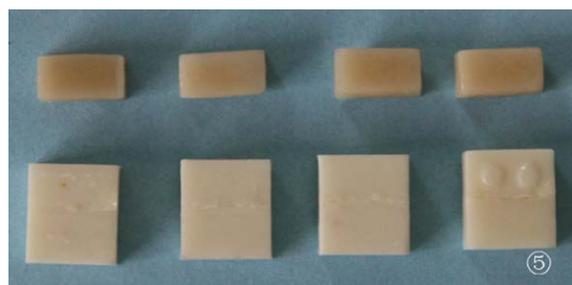


Figure 2: The appearance of fracture surface after shear test.

respectively. Besides, $p < 0.05$ indicates the statistical significance. Hence, the pre-sintering process of bonding porcelain at 1000°C for 15 min was beneficial in improving the bond strength between zirconia framework and veneering porcelain.

The Scanning Electron Microscope (SEM) photo for specimens in Group 2.1 and 2.2 were shown in Figure 3. After sintering, the zirconia layer and porcelain layer for specimens in Group 2.1 were compact and uniform, the sintered interface was smooth and clear, and no obvious pore has been detected. As for specimens in Group 2.2, the

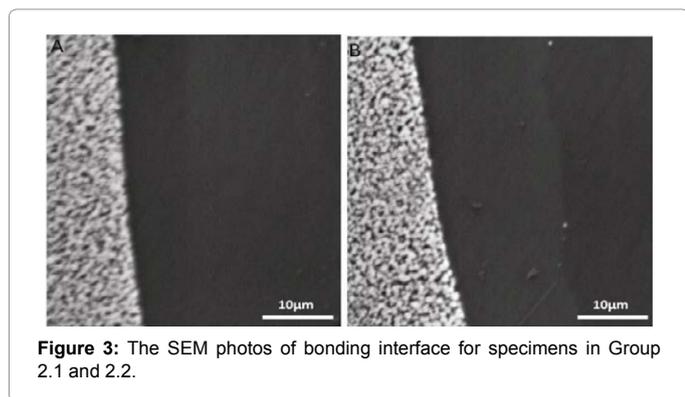


Figure 3: The SEM photos of bonding interface for specimens in Group 2.1 and 2.2.

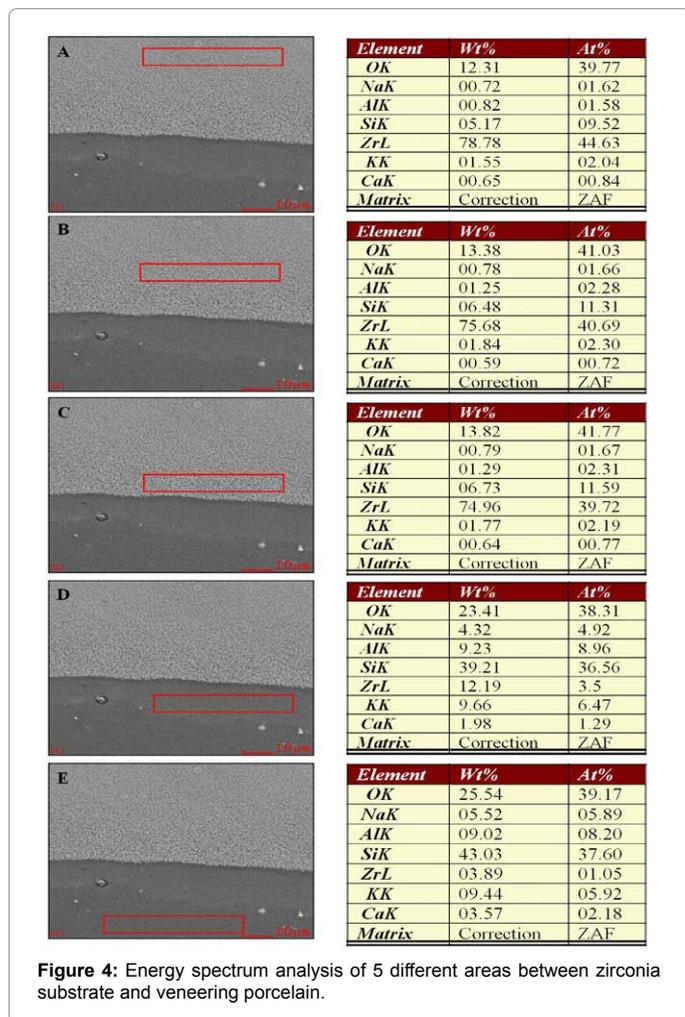


Figure 4: Energy spectrum analysis of 5 different areas between zirconia substrate and veneering porcelain.

Element	A (wt%)	B (wt%)	C (wt%)	D (wt%)	E (wt%)
OK	12.31	13.38	13.82	23.41	25.54
NaK	0.72	0.78	0.79	4.32	5.52
AlK	0.82	1.25	1.29	9.23	9.02
SiL	5.17	6.48	6.73	39.21	43.03
ZrK	78.78	75.68	74.96	12.19	3.89
KK	1.55	1.84	1.77	9.66	9.44
CaK	0.65	0.59	0.64	1.98	3.57

Table 3: Mass percentage of element content in different areas between the veneering porcelain and zirconia substrate.

Element	A (At%)	B (At%)	C (At%)	D (At%)	E (At%)
OK	39.77	41.03	41.77	38.31	39.17
NaK	0.62	1.66	1.67	4.92	5.89
AlK	1.58	2.28	2.31	8.96	8.2
SiL	9.52	11.31	11.59	36.56	37.6
ZrK	44.63	40.69	39.72	3.5	1.05
KK	2.04	2.3	2.19	6.47	5.92
CaK	0.84	0.72	0.77	1.29	2.18

Table 4: Atom percentage of element content in different areas between the veneering porcelain and zirconia substrate.

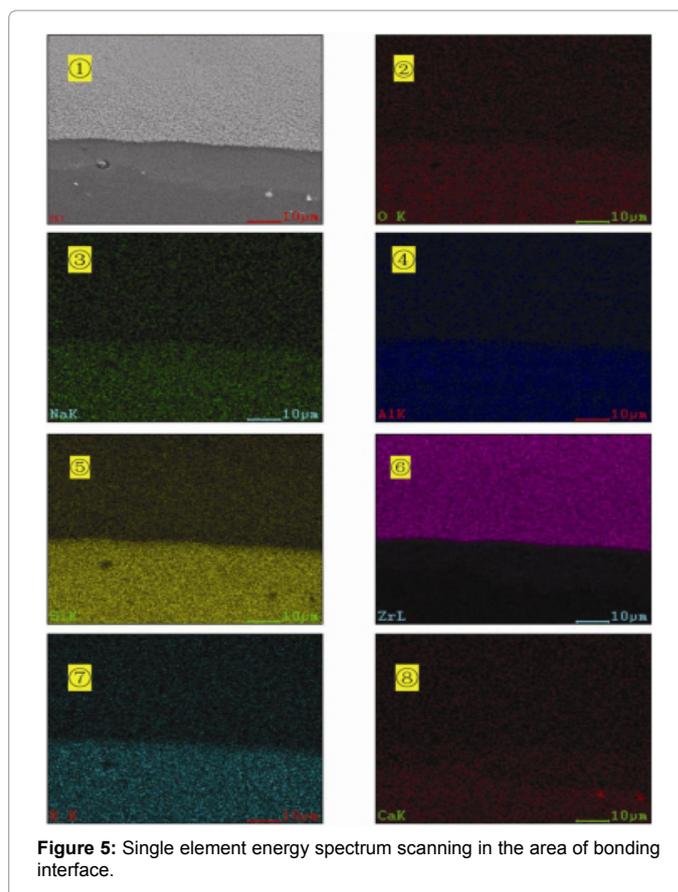


Figure 5: Single element energy spectrum scanning in the area of bonding interface.

zirconia layer, pre-sintered bonding porcelain layer, and the veneering porcelain layer presented a typical triple layer structure.

A specimen from Group 2.2 was selected for the EDS analysis. As shown in Figure 4, the element contents of 5 different areas (A, B, C, D, and E) which were distributed in both sides of the bonding interface, scanned and analyzed. The mass percentage and atom percentage of

the certain element in different areas were listed in Tables 3 and 4, respectively. In addition, the energy-spectrum scanning picture of every single element was illustrated in Figure 5. The element content of Zr in the outer side of zirconia substrate (Area A) was 78.78 wt%, and was slightly reduced to 74.96 wt% in the bonding interface (Area C). Besides, Zr contents were detected to be 12.19 wt% and 3.89% in the bonding porcelain layer (Area D) and the veneering porcelain layer (Area E), respectively. The element content of Si in the veneering porcelain layer (Area E) was 43.03%, and it decreased to be 39.21% in the bonding porcelain layer (Area D). Besides, small amounts of element Si were detected both in the bonding interface (Area C) and zirconia substrate (Areas A and B), indicating the diffusion of Si occurred in the zirconia matrix after pre-sintering process. The chemical bonding was confirmed by element diffusion phenomenon, and it was beneficial in the increasing of bonding strength between zirconia and veneering porcelain.

Effect of sintering temperature on the bond strength

To investigate the effect of sintering temperature on the bond strength, 11 different sintering schedules were performed on 11 groups of specimens. As shown in Table 5, the bond strength was enhanced with the increasing of sintering temperature, and reached to peak value at 960°C. After then, a declination occurred with the continually increasing of sintering temperature. The p-value between each two groups from Group 3.1 to 3.11 was listed in Table 6.

Group No. (Temperature)	Maximum (MPa)	Minimum (MPa)	Average (MPa)	Standard deviation
3.1 (900°C)	39.84	32.16	35.55	3.28
3.2 (910°C)	41.55	34.23	36.71	3.78
3.3 (920°C)	43.57	34.88	37.92	3.68
3.4 (930°C)	46.79	36.28	40.74	5.10
3.5 (940°C)	48.04	38.53	42.98	4.86
3.6 (950°C)	49.08	38.19	43.28	5.07
3.7 (960°C)	50.95	39.22	44.35	4.58
3.8 (970°C)	48.01	37.52	42.77	4.91
3.9 (980°C)	46.33	36.06	40.02	4.11
3.10 (990°C)	42.57	34.75	37.32	3.76
3.11 (1000°C)	39.38	32.99	35.18	3.09
Value of F				16.88*

*: significant difference (p<0.05)

Table 5: Shear strength of specimen in different sintering temperatures.

Group No.	1.1	1.2	1.3	1.4	1.5	1.6	1.7	1.8	1.9	1.10	1.11
1.1	-	0.21	0.035	0.041	0.01	0.009	0.008	0.008	0.028	0.033	0.19
1.2	ns	-	0.42	0.047	0.036	0.007	0.009	0.041	0.025	0.072	0.31
1.3	*	ns	-	0.034	0.029	0.011	0.015	0.036	0.027	0.46	0.035
1.4	*	*	*	-	0.044	0.038	0.023	0.034	0.58	0.032	0.047
1.5	**	*	*	*	-	0.61	0.045	0.54	0.043	0.038	0.025
1.6	**	**	*	*	ns	-	0.039	0.73	0.049	0.008	0.009
1.7	**	**	*	*	*	*	-	0.041	0.044	0.01	0.013
1.8	**	*	*	*	ns	ns	*	-	0.035	0.026	0.017
1.9	*	*	*	ns	*	*	*	*	-	0.042	0.039
1.10	*	*	*	*	*	**	**	*	*	-	0.046
1.11	*	*	*	*	*	**	*	*	*	*	-

ns: no significant difference (p>0.05); *: significant difference (p<0.05); **: very significant difference (p<0.01).

Table 6: p-value between each two groups from Groups 3.1 to 3.11.

Conclusion

The average shear strength for specimens with surface morphology of vertical striations, parallel striations, and humps, were measured to be 27.9 MPa, 27.5 MPa, and 37.2 MPa, respectively, demonstrating that humps in the surface of zirconia substrate remarkably enhanced the bond strength between zirconia substrate and veneering porcelain. The pre-sintering process of bonding porcelain was beneficial in improving the bond strength between zirconia and veneering porcelain. A typical triple layer structure of the zirconia layer, pre-sintered bonding porcelain layer, and the veneering porcelain layer was observed by SEM, and an element penetration phenomenon was confirmed by EDS. The result indicated that an element binding force, which was produced in pre-sintering process, made contribution to be increasing of bond strength. Besides, the bond strength could be enhanced with the increasing of sintering temperature, and reached to peak value at 960°C.

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