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Effect of surface condition of dental zirconia ceramic (Denzir) on bonding

(Running title: SURFACE CONDITION OF ZIRCONIA BONDING)

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Abstract

Yttria partially-stabilized zirconia (YPSZ) ceramics are suitable for dental and medical use because of their high fracture toughness and chemical durability. The purpose of this study was to examine the bonding of a dental YPSZ ceramic (Denzir). After being subjected to various surface treatments the Denzir specimens were bonded to each other using an adhesive resin composite, glass ionomer or zinc phosphate cement. The bonding strength was then determined by the shearing test. No significant difference ($p > 0.05$) was observed between SiC- and Al₂O₃-blasted specimens. In all surface treatments, the shear bonding strength significantly ($p < 0.05$) increased in the order of adhesive resin composite cement > glass ionomer cement > zinc phosphate cement. Silanization with methacryloxy propyl trimethoxysilane slightly increased the bonding strength of the adhesive resin composite.

Keywords: Zirconia, Bonding strength, Surface treatment

Introduction

In recent years the demand for dental restorations with more aesthetic and biocompatible properties has increased and various ceramics have been widely studied and applied as materials for dental restorations¹⁻⁶. Yttria partially stabilized zirconia (YPSZ) is a ceramic that exhibits superior fracture toughness and chemical durability compared to other ceramics^{1, 2}. Because of these suitable properties, YPSZ has been extensively employed for medical and dental uses²⁻⁵. However, manufacturing of dental restorations requires complicated machining of various shapes and dimensions and it is difficult to achieve accurate machining. Through recent progress in the CAD/CAM-technique, YPSZ ceramics can, however, now be employed for complicated dental restorations such as inlays, copings and fixed partial denture (FPD) frameworks⁶⁻¹⁰. Dental restorations using YPSZ are made either by milling enlarged restorations out of homogenous ceramic green-body blanks of zirconia, which are then sintered and shrunk to the desired final dimensions⁷, or by milling the restorations directly with the final dimensions out of highly dense sintered prefabricated YPSZ blanks, called hot isostatic pressed (HIPed) zirconia blanks^{6, 8-10}.

In an earlier report, the authors determined the cytotoxicity and bonding property of HIPed YPSZ¹¹. In that study¹¹, HIPed YPSZ showed no cytotoxicity and exhibited high bonding to glass ionomer cement, but relatively low bonding strength to adhesive resin composite cement¹¹. Kern et al., however, reported that a durable bond to YPSZ was achieved by using a phosphate monomer containing resin composite bonding cement¹². In their study¹², as in most of the earlier studies evaluating the bond strengths of various cementing agents to YPSZ ceramics^{e.g. 13-19}, specimens made out of homogenous ceramic green zirconia were used. Since HIPed YPSZ blanks have been successfully employed in the fabrication of dental all-ceramic crowns and FPDs⁸⁻¹⁰, the bonding properties of this type of zirconia ceramic are of interest. However, in a survey of the literature, only 1

article¹¹⁾ was found that addressed the bonding properties of HIPed YPSZ. The purpose of this study, therefore, was to evaluate the bonding properties of various bonding cements to HIPed YPSZ and the effects of surface roughness and pretreatments on bonding strength.

Materials and methods

Twenty HIPed YPSZ (Denzir, Cad.esthetics AB, Skellefteå, Sweden) specimens were machined into a rectangular shape (14 mm x 14 mm x 5 mm) using the Cad.esthetics CAD/CAM system (Cad.esthetics AB). Some of these specimens were then sandblasted with 70 μm Al_2O_3 (Hi-Alumina, Shofu, Kyoto, Japan) and the remainder with 125 μm SiC powder (Carborundum, Shofu, Kyoto, Japan) with 0.3 MPa for 30 sec at a distance of 10 mm. Thereafter, SiC-blasted specimens were etched with a phosphoric acid gel (K-etchant gel, Kuralay, Kurashiki, Japan) for 10 seconds. In addition, the etched specimens intended to be cemented with an adhesive resin composite cement were then silanized by either: *i*) treatment with Clearfil Porcelain Bond Activator (Kuraray, Kurashiki, Japan) mixed with Clearfil Linerbond IIS (Kuraray, Kurashiki, Japan) for 10 sec in accordance with the manufacturers' instructions, or *ii*) heating with 10% methacryloxy propyl trimethoxysilane (MPTS, Tokyo Kasei Kogyo, Tokyo, Japan)-toluene (dehydrated) solution at 60°C for 1 hour and drying in a vacuum.

The cements used for the bonding test were *i*) a zinc phosphate cement (Elite Cement, GC, Tokyo, Japan), *ii*) a glass ionomer cement (Fuji I, GC, Tokyo, Japan) and, *iii*) an adhesive resin composite cement (Panavia 21, Kuraray, Kurashiki, Japan). Using one of the above-mentioned cements, the surface-treated specimens were bonded to corresponding specimens with a similar surface condition. The bonding area was 14mm x 5mm. The bonded specimens were then stored at 37°C, 100% RH for 1 hour. Seven replicates were prepared for each condition. The abbreviations of surface treatments and cements are tabulated in Table 1.

Table 1

The surface profile and mean surface roughness (Ra) were determined using a

surface profilometer (Surfcom 2000, Tokyo Seimitsu Co.Ltd., Tokyo, Japan).

The bonding strength was determined by the shearing test method using a universal testing machine (Model 4204, Instron, Canton, U.S.A.). The apparatus for shear test is shown in Fig. 1. The shear force was applied at cross-head speed of 0.5mm/sec. The results of the bonding test were analyzed statistically using ANOVA and Scheffe's test at a significance level of $p < 0.05$. After the bonding test, the YPSZ blocks were polished with emery paper (#80) until the attached cement layer was removed. Then, the polished blocks were surface treated again and used for the next bonding test.

Fig. 1

After the shear test, the fracture surfaces were analyzed using a scanning electron microscope (SEM) (S-2380, Hitachi, Tokyo, Japan). With an energy dispersed X-ray detector (EDX) (Genesis, EDAX Japan, Tokyo, Japan) the elemental distribution image of the observed surface was determined.

Results

Figure 2 shows the surface profiles and the mean surface roughness level (Ra) of Al₂O₃- and SiC-blasted and phosphoric acid-etched YPSZ specimens. The SiC-blasted surfaces had a clearly rougher profile than the Al₂O₃-blasted surfaces. The mean surface roughness (Ra) of the SiC-blasted surface was 0.86 μm, twice that of the Al₂O₃-blasted surface (0.43 μm).

Fig. 2

The shear bonding strengths of the Al₂O₃- and SiC-blasted and phosphoric acid-etched YPSZ specimens cemented with the three types of cement are shown in Fig. 3. In all surface treatments, the shear bonding strength increased in the order of adhesive resin composite cement > glass ionomer cement > zinc phosphate cement. Among the three types of cement, the bonding strength differed significantly ($p < 0.05$). Between the Al₂O₃- and SiC-blasted YPSZ specimens there was no significant difference ($p > 0.05$) in the shear bond strengths obtained. Thus, the bonding strength depended on the type of cement, and the surface roughness did not significantly affect the bonding strength.

Fig. 3

Figure 4 shows the effects on the bonding strength of surface treatments using phosphoric acid, PBA or MPTS in combination with the various cements. The significance of difference is tabulated in Table 2. In all surface treatments, the shear bonding strength increased in the order of adhesive resin composite cement > glass ionomer cement > zinc phosphate cement. Among the adhesive resin cement, The MPTS treatment showed highest bonding strength but their difference was in low significance ($0.1 > p > 0.05$).

Fig. 4

Table 2

Figure 5 shows the SEM and elemental distribution images of the fracture surfaces bonded with zinc phosphate, glass ionomer and adhesive resin composite

Fig. 5

cements. The surfaces of the zinc phosphate cemented specimens were clearly separated into a cement part (Zn rich area) and a YPSZ surface (Zr-rich area). The cement/YPSZ boundary was clear and interface fracture between the YPSZ and zinc phosphate cement was suggested. In contrast, the glass ionomer- and adhesive resin composite cement-bonded specimens showed complex, intricate surfaces, which implied partly cohesive fracture.

Discussion

In this study, the effects on the shear bond strength of various surface conditions of YPSZ in combination with various cements were studied. The bonding strength of YPSZ with two different surface roughnesses and three types of cement was tested. The different surface roughnesses studied did not influence the bonding strength, whereas the different types of silanization and cement used significantly affected the bonding strength of the adhesive resin composite cement. In all surface pretreatments, the shear bonding strength increased in the order of adhesive resin composite cement > glass ionomer cement > zinc phosphate cement.

In a previous report, the authors determined the bonding strength of YPSZ using threaded and tapered holes in a brass plate and YPSZ milled specimens.¹¹⁾ In that study¹¹⁾, the glass ionomer cement had the highest bonding strength, whereas no significant difference was seen between adhesive resin composite and zinc phosphate cements.¹¹⁾ In contrast, in the present study the adhesive resin composite cement showed the highest bonding strength. Differences in the test methods are considered to be one possible cause for the different results obtained in the studies. In the previous study¹¹⁾, the specimens and the brass plate were tapered. Therefore, the applied force probably included not only shearing force but also tensile force. In addition, when luted with the various cements used, the YPSZ specimens were placed manually in drilled tapered holes.¹¹⁾ This may have resulted in cement layers whose thickness was not constant. With the present method, the flat surface of the YPSZ specimen was bonded to another surface with a similar surface condition, which should reproduce the bonding condition more consistently than the previous method. In the present study, the shearing force was applied in the direction perpendicular to the bonding plane. The present method should, therefore, more

accurately determine the bonding strength to YPSZ of the three types of cement studied.

The MPTS treatment with the adhesive resin cement showed the higher shear bonding strength than the other treatment, but their difference was in low significance ($0.1 > p > 0.05$). The reason for the difference of effectiveness between MPTS and PBA is considered to be as follows. In the PBA treatment, a mixture of PBA and the primer (Clearfil Linerbond IIS) was applied on the YPSZ surface and air-dried. In contrast, the MPTS treatment was carried out at 60°C and drying was done in a vacuum after removal of the residual MPTS/toluene solution. Therefore, the formed surface layer would be thinner and more tightly bonded to the YPSZ surface than when using the PBA pretreatment. Thus, the results in the present study indicated that the adhesive resin composite cement in combination with MPTS pretreatment bonded more effectively to the YPSZ than in combination with the PBA treatment.

Values reported for the bonding strength of various adhesive resin-cementing agents to zirconia ceramic specimens in previous studies were between 1.7 MPa and 70.4 MPa.^{e.g. 17, 20} In most of the studies that addressed the bonding strength to zirconia ceramics, the specimens were made of homogenous ceramic green-body blanks of zirconia, and various bonding systems and pretreatments were used.^{e.g. 13-19} In the current study, specimens of highly dense sintered prefabricated zirconia blanks, called hot isostatic pressed (HIPed) zirconia blanks, were used. In an earlier report on the bonding strength of HIPed YPSZ zirconia¹¹), the mean values were 12 MPa for adhesive resin composite cement, 21 MPa for glass ionomer cement and 9 MPa for zinc phosphate cement. That is, the mean value of the resin composite cement was close to the value obtained in the current study, whereas the values obtained for the zinc phosphate and

glass ionomer cements were lower.

The effects of surface treatments on bonding to dental ceramics other than zirconia have also been presented in earlier studies.^{12, 21-24)} The shear bonding strengths when using adhesive resin composite cements and silanated surfaces of a lithium disilicate glass-ceramic and feldspar ceramics have been reported to be 47 MPa for Empress2¹²⁾ (Ivoclar Vivadent, Schaan, Lichtenstein), 18 MPa for Vita VMK 68²²⁾, 42 MPa for Cerec 2 Vitablock Mark II²³⁾ and 55 MPa for Vita Celay²⁴⁾ (all Vita products from Vita Zahnfabrik, Bad Säckingen, Germany). Aida et al. reported that the bonding strength of another feldspar ceramic (Laminabond Porcelain, Shofu, Kyoto, Japan) increased with silanization using commercial priming silane agents other than MPTS²¹⁾. Those results partly contradict the results obtained in the present study. The reason could be differences in the silane treatment and the ceramics studied. In this context it should be noted that hydrofluoric acid etching of conventional silica-based ceramics usually improves the effect of silanization and adhesive bonding¹²⁾, whereas dental zirconia ceramics are unetchable with hydrofluoric acid. However, it should also be pointed out that it has been said that the phosphate ester group in the Panavia 21 resin composite directly bonds to metal oxides²⁶⁾ and bonds chemically to zirconia ceramics.¹²⁾

The reported bonding strengths of cements to dental ceramics, thus, vary within a wide range and assessment of their clinical significance is difficult. It has been suggested that 10-13 MPa is the minimum strength needed for clinical bonding^{27, 28)} On the other hand, the in vitro bond strengths to acid-etched human dentin of various commercial resin composite bonding cements, which have been in clinical use for a relatively long time, are reported to range from 1.1 MPa to 14.8 MPa.²⁹⁾ For conventional zinc phosphate cement, Øilo³⁰⁾ reported a tensile bond strength to dentin of 0.6 MPa and Richardsson et al. 0.9 MPa³¹⁾ Although those values seem to be very low, and are

considerably inferior to those suggested as the acceptable minimum strength for clinical bonding^{27, 28)}, zinc phosphate cements have been successfully used clinically for a very long time to lute cast dental restorations. To assess the clinical performance of bonding systems, in vitro studies should, therefore, be supplemented with clinical studies with long follow-up times.

In most of the earlier studies dealing with zirconia specimens, various surface pre-treatments such as sandblasting or tribochemical methods for silicating the surfaces were used^{e.g. 13-19)} However, mechanical treatments of zirconia should be done with caution because it has been demonstrated that heat treatment, sandblasting and grinding can influence its mechanical properties^{10, 32, 33)} In a recently presented study by Sundh and Sjögren it was stated that the effect on the fracture resistance of zirconia depended on, among other things, the time the specimens were subjected to sandblasting.¹⁰⁾ This is probably because sandblasting treatment and/or grinding can induce compressive stresses and/or phase transformation on the surface, which increases the strength, but also can induce flaws and other defects that reduce strength. Thus, to find the best possible technique more studies are needed to determine the effects of surface treatments on the bond strength and mechanical properties of zirconia ceramics.

Conclusion

Within the limitations of this in vitro study the following conclusions were drawn:

- 1 The different surface roughnesses obtained between SiC- and Al₂O₃-blasted specimens did not significantly affect the bonding strength.
- 2 In all surface pretreatments, the shear bonding strength significantly increased in the order of adhesive resin composite cement > glass ionomer cement > zinc phosphate cement.

- 3 The adhesive resin composite cement bonds slightly better to YPSZ in combination with 10% methacryloxy propyl trimethoxysilane-toluene (dehydrated) solution than the other surface treatment.

Acknowledgements

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References

- 1 Green DJ, Hannink RHJ, Swain MV. Transformation Toughening of Ceramics, CRC Press, Boca Raton, 1989, pp, 137-144,
- 2 Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. *Biomaterials* 1999; 20: 1-25.
- 3 Cales B, Stefani Y, Lilley E. Long-term *in vivo* and *in vitro* aging of a zirconia ceramic used in orthopaedy. *J Biomed Mater Res* 1997; 28: 619-624.
- 4 Koutayas SO, Kern M. All-ceramic posts and cores: The state of the art. *Quintessence Int* 1999; 30: 383-392.
- 5 Akagawa Y, Ichikawa Y, Nikai H, Tsuru H. Interface histology of unloaded and early loaded partially stabilized zirconia endosseous implant in initial bone healing. *J Prosthet Dent* 1993; 69: 599-604.
- 6 Sjölin R, Sundh A, Bergman M. The Decim system for the production of dental restorations. *Int J Computerized Dent* 1999; 2: 197-207.
- 7 Filser F, Kocher P, Weibel F, Luthy H, Scharer P, Gauckler LJ. Reliability and strength of all-ceramic dental restorations fabricated by direct ceramic machining (DCM). *Int J Comput Dent* 2001; 4: 89-106.
- 8 Sundh A, Sjögren G. A comparison of fracture strength of yttrium-oxide-partially-stabilized zirconia ceramic crowns with varying core thickness, shapes and veneer ceramics. *J Oral Rehabil* 2004; 31: 682-688.
- 9 Sundh A, Molin M, Sjögren G. Fracture resistance of yttrium oxide partially-stabilized zirconia all-ceramic bridges after veneering and mechanical fatigue testing. *Dent Mater* 2005; 21: 476-482.
- 10 Sundh A, Sjögren G. Fracture resistance of all-ceramic zirconia bridges with differing phase stabilizers and quality of sintering. *Dent Mater*, in press

- 11 Uo M, Sjögren G, Sundh A, Watari F, Bergman M, Lerner U. Cytotoxicity and bonding property of dental ceramics. *Dent Mater* 2003; 19: 487-492.
- 12 Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. *Dent Mater* 1998; 14: 64-71.
- 13 Blatz MB, Sadan A, Martin J, Lang B. In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling. *J Prosthet Dent* 2004; 91: 356-362.
- 14 Piwowarczyk A, Lauer HC, Sorensen JA. The shear bond strength between luting cements and zirconia ceramics after two pre-treatments. *Oper Dent* 2005; 30: 382-388.
- 15 Ernst CP, Cohnen U, Stender E, Willershausen B. In vitro retentive strength of zirconium oxide ceramic crowns using different luting agents. *J Prosthet Dent* 2005; 93: 551-558.
- 16 Derand T, Molin M, Kvam K. Bond strength of composite luting cement to zirconia ceramic surfaces. *Dent Mater* 2005; 21: 1158-1162.
- 17 Lüthy H, Loeffel O, Hammerle CH. Effect of thermocycling on bond strength of luting cements to zirconia ceramic. *Dent Mater* 2006; 22: 195-200.
- 18 Matinlinna JP, Heikkinen T, Ozcan M, Lassila LV, Vallittu PK. Evaluation of resin adhesion to zirconia ceramic using some organosilanes. *Dent Mater*, in press
- 19 Wolfart M, Lehmann F, Wolfart S, Kern M. Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods. *Dent Mater*, in press
- 20 Ernst CP, Cohnen U, Stender E, Willershausen B. In vitro retentive strength of zirconium oxide ceramic crowns using different luting agents. *J Prosthet Dent* 2005; 93: 551-8.

- 21 Aida, M, Hayakawa T, Mizukawa K. Adhesion of composite to porcelain with various surface conditions, *J Prosthet Dent* 1995; 73: 464-70.
- 22 Matsumura H, Kato H, Atsuta M. Shear bond strength to feldspathic porcelain of two luting cements in combination with three surface treatments. *J Prosthet Dent* 1997; 78: 511-517.
- 23 Kamada K, Yoshida K, Atsuta M. Effect of ceramic surface treatments on the bond of four resin luting agents to a ceramic material. *J Prosthet Dent* 1998; 79: 508-513.
- 24 Bayindir YZ, Bayindir F, Akyil SM. Bond strength of permanent cements in cementing cast to crown different core build-up materials. *Dent Mater J* 2004; 23: 117-120.
- 25 Ide T, Tanoue N, Yanagida H, Atsuta M, Matsumura H. Effectiveness of bonding systems on bonding durability of a prefabricated porcelain material. *Dent Mater J* 2005; 24 : 257-260.
- 26 Wada T. Development of a new adhesive material and its properties. *Proceedings of the International Symposium on Adhesive Prosthodontics*. Amsterdam, Netherlands, Chicago: Academy of Dental Materials; 1986, p. 9-18.
- 27 Kappert HF, Krah M. Keramiken—eine Übersicht. *Quintessenz Zahntech* 2001; 2: 668-704.
- 28 Thurmond J, Barkmeier W, Wildweding M. Effect of porcelain surface treatments on bond strength of composite resin bonded to porcelain. *J Prosthet Dent* 1994; 72: 355-359.
- 29 Piwowarczyk A, Bender R, Ottl P, Lauer HC. Long-term bond between dual-polymerizing cementing agents and human hard dental tissue. *Dent Mater*, in press
- 30 Øilo G. Adhesive bonding of dental luting cements; influence of surface treatment. *Acta Odontol Scand* 1978; 36: 263-270.

- 31 Richardson DW, Tao L, Pashley DH. Bond strengths of luting cements to potassium oxalate-treated dentin. *J Prosthet Dent* 1990; 63: 418-422.
- 32 Zhang Y, Lawn BR, Rekow ED, Thompson VP. Effect of sandblasting on the long-term performance of dental ceramics. *J Biomed Mater Res B Appl Biomater* 2004; 15: 381-386.
- 33 Guazzato M, Quach L, Albakry M, Swain MV. Influence of surface and heat treatments on the flexural strength of Y-TZP dental ceramic. *J Dent* 2005; 33: 9-18.

Figure Captions

Figure 1 Apparatus for the shear bonding test.

Figure 2 Surface profiles and mean surface roughness (Ra) of YPSZ specimens blasted with Al_2O_3 or SiC.

Figure 3 The shear bonding strength of Al_2O_3 - and SiC-blasted and phosphoric acid-etched YPSZ in combination with the three cements studied.

Figure 4 The effect on the bonding strength to SiC-blasted and phosphoric acid-etched YPSZ specimens of various surface treatments in combination with the three cements studied.

Figure 5 SEM and elemental distribution images of fractured surfaces bonded with various cements. (C: cement, Z: YPSZ)

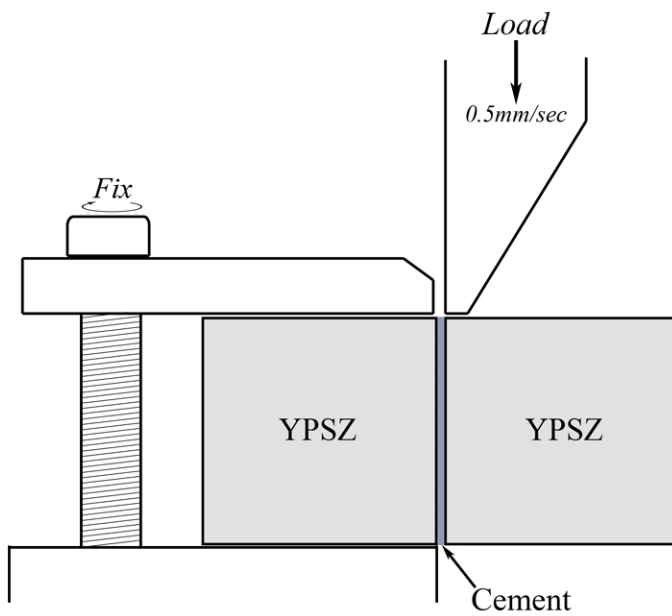
Table 1 The abbreviations for surface conditions and cements.

Abbreviations	Cements or surface treatments
ZP	zinc phosphate cement
GI	glass ionomer cement
AR	adhesive resin composite cement
AB	Al ₂ O ₃ -blasted
SB	SiC-blasted
PE	phosphate gel-etched
PBA	treated with Clearfil Porcelain Bond Activator
MPTS	treated with 10% methacryloxy propyl trimethoxysilane-toluene solution

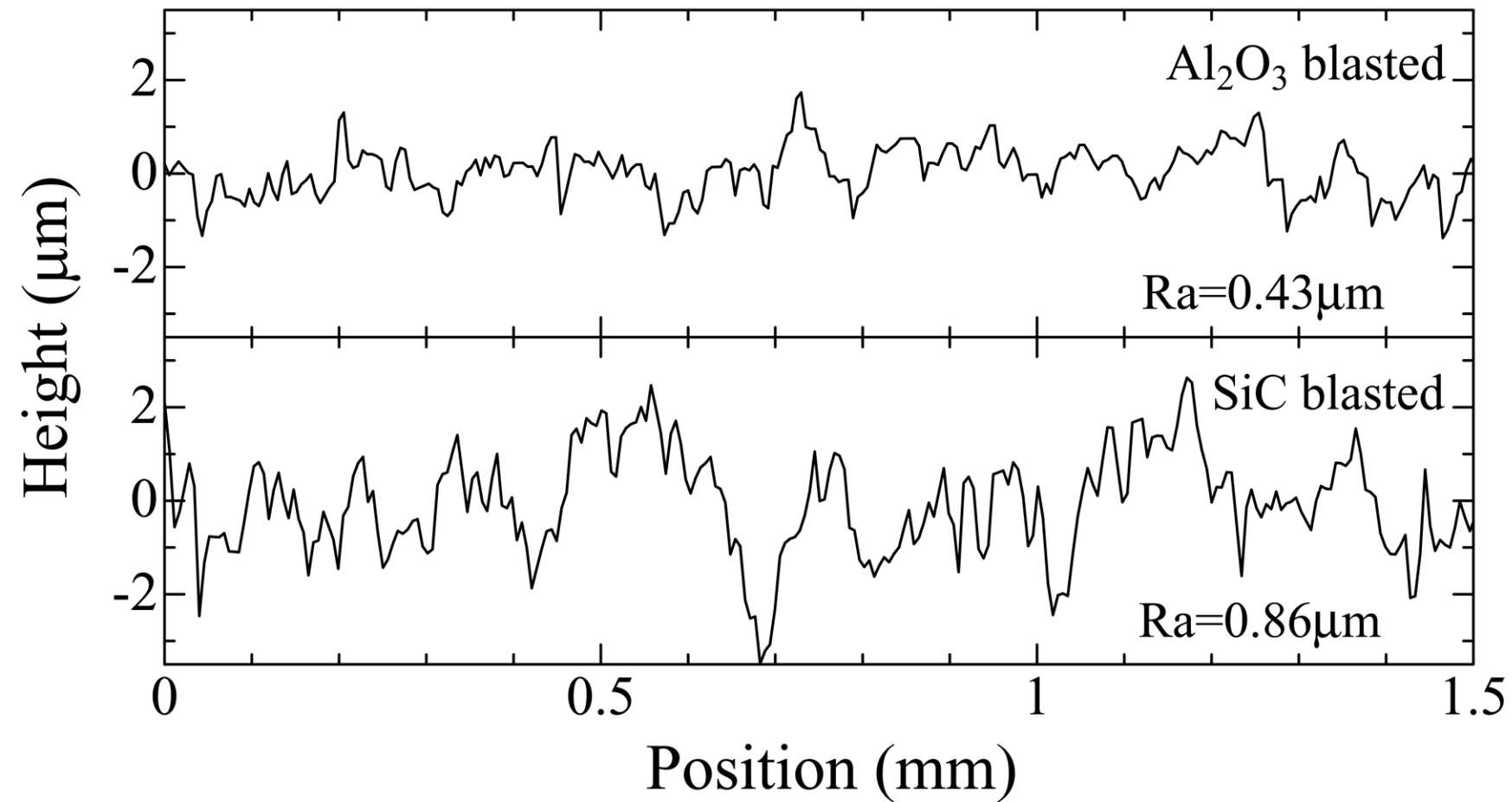
Table 2 Summary of statistical analysis of the bond strength of the SiC blasted specimens.

Results of ANOVA supplemented with Scheffe's test. (n.s.: no significant difference, *: $p < 0.05$, **: $p < 0.001$)

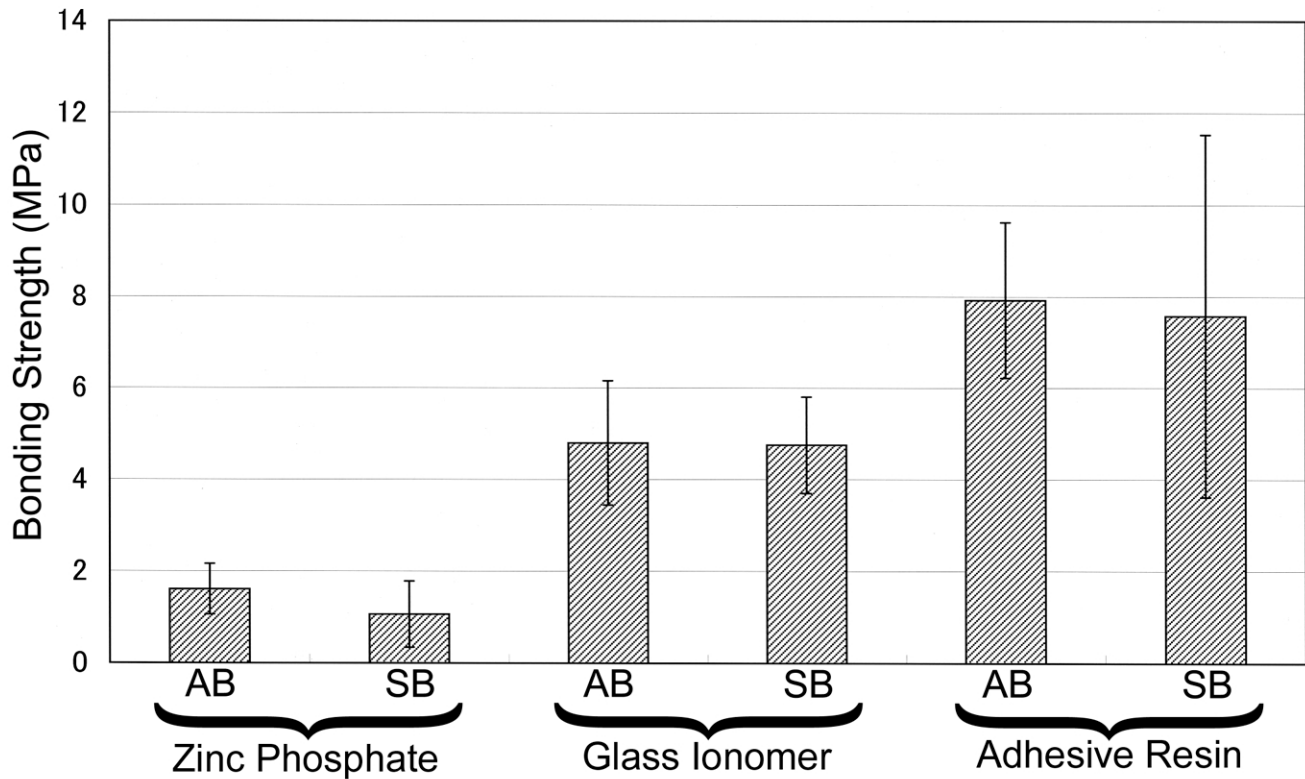
Cements		ZP		GI		AR			
Cements	surface treatment	SB	SB PE	SB	SB PE	SB	SB PE	SB PE PBA	SB PE MPTS
ZP	SB SB-PE	n.s.							
GI	SB SB-PE	**	**	n.s.					
AR	SB	*	*	n.s.	n.s.				
	SB-PE	*	*	*	n.s.	n.s.			
	SB-PE-PBA	**	**	**	**	n.s.	n.s.		
	SB-PE-MPTS	**	**	**	*	n.s.	n.s.	n.s.	



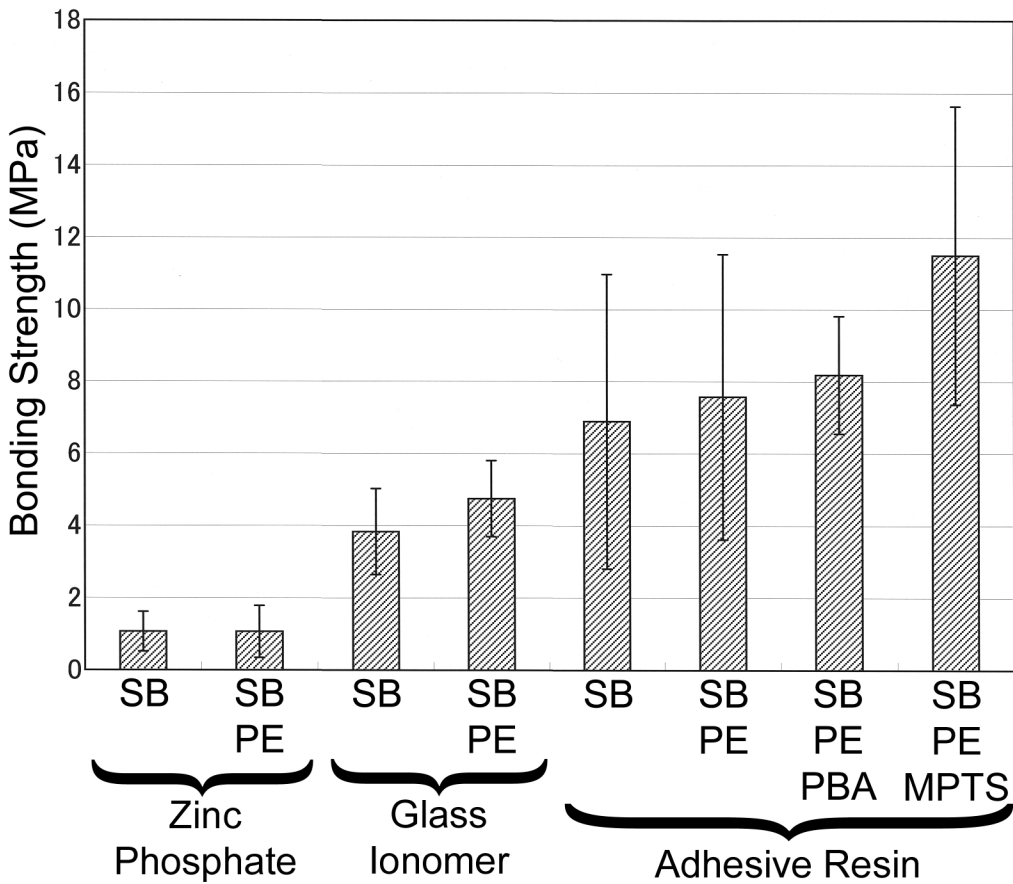
(Fig.1)



(Fig.2)

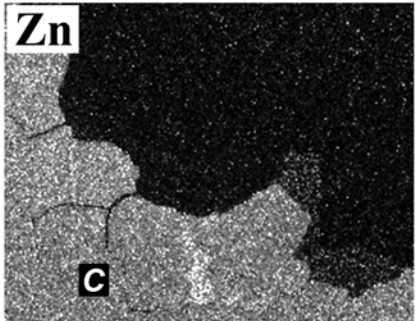
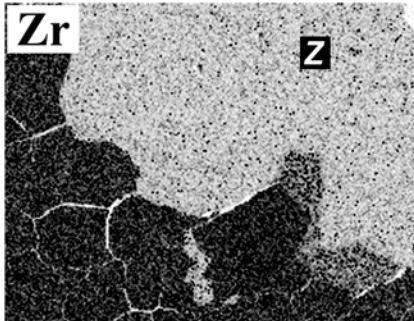


(Fig.3)

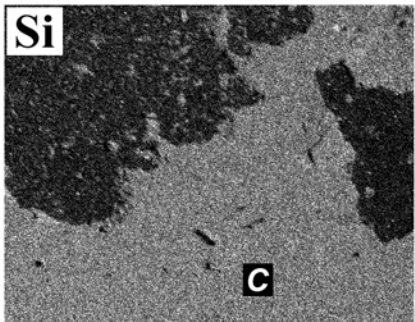
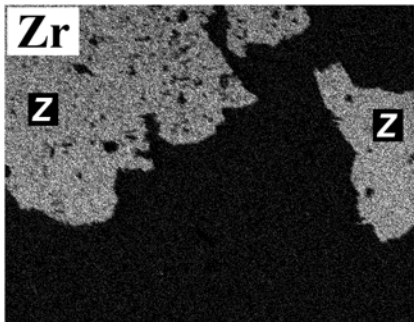
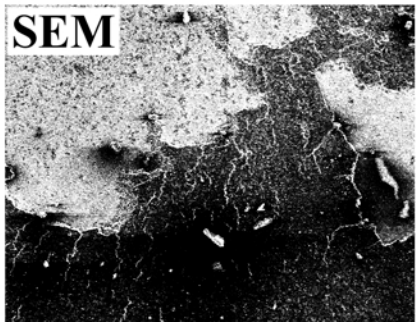


(Fig.4)

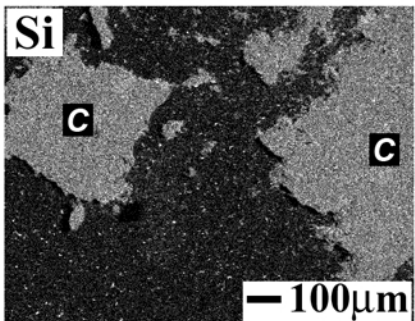
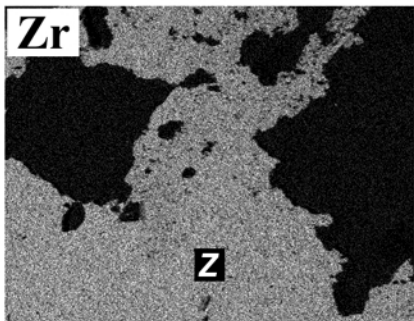
Zinc
Phosphate



Glass
Ionomer



Adhesive
Resin



(Fig.5)