ORIGINAL ARTICLE

J Korean Dent Sci. 2014;7(2):49-57
http://dx.doi.org/10.5856/JKDS.2014.7.2.49
ISSN 2005-4742

Effect of Different Surface Treatment on the Shear Bond Strength between Yttria-Tetragonal Zirconia Polycrystal and Non-10-Methacryloyloxydecyl Dihydrogen Phosphate-Containing Resin Cement

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Purpose: To evaluate the effect of different surface treatment methods (yttria-tetragonal zirconia polycrystal [Y-TZP] primers, air-abrasion, and tribochemical surface treatment) on the shear bond strength between (Y-TZP) ceramics and etch-and-rinse non-10-methacryloyloxydecyl dihydrogen phosphate (MDP)-containing resin cements.
Materials and Methods: Y-TZP ceramic surfaces were ground flat with 600-grit silicone carbide abrasives paper and then divided into seven groups of ten. They were treated as the following: untreated (control), Monobond Plus (IvoclarVivadent), Z-PRIME Plus (Bisco Inc.), ESPE Sil with CoJet (3M ESPE), air-abrasion, Monobond Plus with air-abrasion, and Z-PRIME Plus with air-abrasion. The surface of Y-TZP specimens was analyzed under a scanning electron microscope (SEM). Non-MDP-containing cements were placed on the surface-treated Y-TZP specimens. After thermocycling, shear bond strength test was performed. Bond strength values were statistically analyzed using one-way analysis of variance and Student-Newman-Keuls multiple comparison test ($P<0.05$).
Result: The Z-PRIME Plus treatment in combination with air-abrasion produced the highest bond strength (14.94±1.70 MPa) followed by Monobond Plus combined with air-abrasion (10.70±1.71 MPa), air-abrasion (10.47±1.60 MPa), ESPE Sil after CoJet treatment (10.38±0.87 MPa), Z-PRIME Plus application (10.00±1.70 MPa), and then Monobond Plus application (9.25±0.86 MPa). The control (6.70±1.49 MPa) indicated the lowest results ($P<0.05$). The SEM results showed different surface morphologies according to surface treatment methods compared with the Y-TZP control.
Conclusion: The shear bond strength between the Y-TZP ceramic and the non-MDP-containing resin cement was the greatest when the surface was treated with air-abrasion and MDP-containing Z-PRIME Plus primer.

Key Words: Bond strength; Primer; Resin cements; Surface treatment; Yttria-tetragonal zirconia polycrystal ceramic

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Received for publication October 22, 2014; Returned after revision November 29, 2014; Accepted for publication December 6, 2014

Introduction

The use of all-ceramic materials is on the rise in dentistry, and this is due to the superior physical properties and esthetics of all-ceramic materials[1,2]. Among the all-ceramic materials, yttria-tetragonal zirconia polycrystal (Y-TZP) is clinically popular because of high fracture toughness and fracture strength, but unlike other silica-based ceramics, it is resistant to hydrofluoric acid-etching and fails to form a reliable and durable bond, which is a critical weakness[3].

Predictable cementation is a key factor in the clinical success of all restorative materials including Y-TZP[4,5]. Resin cements are currently recommended for cementation of ceramic restorations[6]. Two components contribute to such adhesive bonding[7]. First is the micromechanical component, which is achieved by interlocking of the pre-treated roughened surface and resin cement. The second component is the chemical bonding between the two materials. Silane (3-methacryloxyp rophyltrimethoxy silane) is bifunctional as it bonds to both the inorganic component of ceramics and the organic polymers of adhesives[6,8]. One end of silane forms an ionic bond with silica in ceramics, while the other end forms covalent bonds with methacrylate groups in resin[6,8].

However, unlike glass or alumina-based ceramics, a consensus on the cementation method using mechanical and chemical adhesion has not been established with Y-TZP, and it is still controversial[9]. Air-abrasion, which is used for micromechanical interlocking, contributes to increasing the bond strength, but it also causes microcracks and phase transformation[9-11]. In addition, tribochemical method is used because silane application is not effective with Y-TZP, but some claim that the silica-coating forms a weak link with Y-TZP and is not any more effective than air-abrasion without chemical bonding effect[9,12-14]. According to previous studies, air-abrasion contributes to micromechanical bonding, and strong and durable bond strength is achieved when resin cements containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) is used[9,15,16]. In case of resin cements containing MDP, there wasn't any significant difference between multi-step self-etch resin cements and self-adhesive resin cements with one step process[15].

Clinically, dentists often use etch-and-rinse type non-MDP-containing resin cements. Such non-MDP-containing resin cements are known to be effective in bonding glass ceramics and indirect composites[17-19]. However, confused clinicians use various combined approach for bonding Y-TZP, which is not based on any scientific evidence. Due to insufficient data, it is necessary to evaluate the bond strength between Y-TZP ceramic and non-MDP-containing resin cement when either conventional surface treatment methods or the newly developed MDP-containing primers are used.

Therefore, the aim of this study was to evaluate the effect of various surface treatments such as air-abrasion, MDP primer and tribochemical method on the shear bond strength between Y-TZP ceramic and non-MDP-containing resin cements. The null hypothesis tested was that different surface treatment methods do not influence the bond strength to Y-TZP ceramic.

Materials and Methods

1. Specimen Preparation

Partially sintered Y-TZP blocks of 97% zirconium dioxide stabilized with a 3% Yttria-Lava Frame (3M ESPE, St. Paul, MN, USA), 19 mm in diameter and 100 mm in height, were sectioned using a low concentration diamond blade (60-20075 Wafering Blade; Allied High Tech Products Inc., Compton, CA, USA) to obtain 4 mm thick ceramic disks. Using 600-grit silicone carbide abrasives, the surfaces of each specimen were polished and ground under water cooling. The Y-TZP ceramic specimens were cleaned ultrasonically for three minutes in distilled water, and sintered according to the manufacturer's instructions. Afterwards, the specimens were embedded in polyethylene molds with 19 mm inner diameter, 21 mm outer diameter, and 12 mm height. One side of each disk was left exposed for cement bonding.

2. Surface Treatments and Bonding Procedure

Depending on the surface treatment method and the resin cement used, seventy specimens were randomly assigned to seven groups of 10 specimens each (total n=70; n=10 per group). Fig. 1 and Table 1 show the experimental design and the materials used in this study, respectively. The three groups without air-abrasion treatment were treated with either Monobond Plus (IvoclarVivadent, Schaan, Liechtenstein), Z-PRIME Plus (Bisco Inc., Schaumburg, IL, USA), or did not undergo any primer treatment. Of the four remaining groups, three groups were treated with air-abrasion at a standoff distance of 10 mm with a 3.5 bar press for 15 seconds using Al_2O_3 particles of 50 μm grain size. After air-abrasion treatment, the surface was rinsed for 30 seconds and then air-dried for 30 seconds. As with the three groups without air-abrasion,

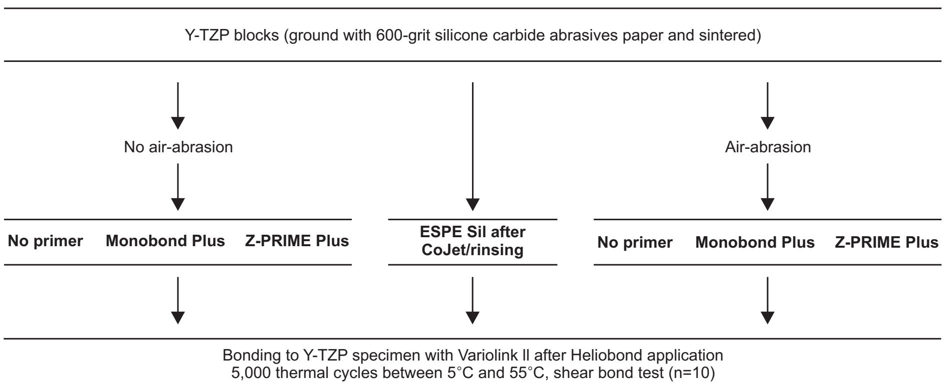

Fig. 1. Experimental design according to surface treatment on yttria-tetragonal zirconia polycrystal (Y-TZP) specimens in this study.

Table 1. Experimental materials and their characteristics

Materials	Brand	Product	Manufacturer (lot #)
Y-TZP	Lava	97% zirconium dioxide stabilized with 3% Yttria-Lava Frame	3M ESPE, St. Paul, MN, USA (386993)
Primer	Monobond Plus	Alcohol solution of silane methacrylate, phosphoric acid methacrylate and sulphide methacrylate	IvoclarVivadent, Schaan, Liechtenstein (M35022)
	Z-PRIME Plus	HEMA, BPDM, ethanol, MDP	Bisco Inc., Schaumburg, IL, USA (900012783)
Silane agent	ESPE Sil	MPS, ethanol	3M ESPE, St. Paul, MN, USA (405022)
Resin cement	Variolink II	Bis-GMA, urethane dimethacrylate, triethylene glycol dimethacrylate, barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide, catalysts, stabilizers, pigments	IvoclarVivadent, Schaan, Liechtenstein (N01551)
Tribochemical silica coating	CoJet	30-μm silica-coated aluminum particles	3M ESPE, St. Paul, MN, USA (368315)

Y-TZP: yttria-tetragonal zirconia polycrystal, HEMA: hydroxyethyl methacrylate, BPDM: biphenyl dimethacrylate, MDP: 10-methacryloyloxydecyl dihydrogen phosphate, MPS: 3-methacryloxyprophyltrimethoxy silane, Bis-GMA: bisphenol A-glycidyl methacrylate.

they were either treated with Monobond Plus or Z-PRIME Plus, or not treated with any primer. The final group was treated with tribochemical silica-coating of 30 μm-sized particles using the CoJet system (3M ESPE). Afterwards, the surface was rinsed for 30 seconds and then air-dried for 30 seconds. A silane primer (ESPE Sil; 3M ESPE) was applied to the dried surface. The examination of surface treatment on the Y-TZP was performed using a scanning electron microscope (SEM, S-4700 FESEM; Hitachi, Tokyo, Japan) under ×3,000. After Heliobond (IvoclarVivadent) was applied to the Y-TZP surface, an etch-and-rinse cement (Variolink II; IvoclarVivadent) was mixed according to the manufacturer's instruction and placed in a #5 sized gel-cap with a 16.8 mm^2 surface area. The gel-cap was placed on the ceramic specimen and light-cured for 20 seconds each from all four sides using a light emitting diode curing light unit (Eliper S10; 3M ESPE) at 600 mW/cm^2. The ceramic specimens were left to polymerize further for 1 hour at 23°C±1°C. Afterwards the specimens were stored in distilled water at 37°C for 23 hours. The specimens were subjected to thermocycling (5°C~55°C for 5,000 cycles). The transfer time between baths was 2 seconds with 30 seconds dwelling time at each temperature.

3. Bond Strength Test and Surface Analysis

The specimens were loaded on the adhesive interface with a jig of the universal testing machine (LF-plus; Ametek Inc., Largo, FL, USA) until a failure occurred at a crosshead speed of 0.5 mm/min. The maximum stress (MPa) required to produce failure was recorded by the computer software. The failure mode was observed under a stereomicroscope (×90).

4. Statistical Analysis

The R programming language (R Foundation for Statistical Computing, Vienna, Austria)[20] was used for data analysis. The normality of data and the equality of variance were checked. A one-way ANOVA and a Student-Newman-Keuls multiple comparison test were performed. The mean difference was regarded as significant at the level of $P<0.05$.

Result

The means and standard deviations for shear bond strength of all groups are presented in Table 2. One-way ANOVA was used to calculate the statistical significance for the surface treatments ($P<0.05$). Air-abrasion, the use of a primer and CoJet treatment were all more effective than the control group treatment. The Z-PRIME Plus treatment after air-abrasion group showed the best result.

The failure mode distribution for all samples is shown in Fig. 2. The results showed a high

Table 2. Shear bond strength results (MPa) of samples using Variolink II resin cement with different surface treatment and priming condition on Y-TZP

Priming conditions	Surface conditions	
	No air-abrasion (polished)	Air-abrasion
None	6.70±1.49^{a}	10.47±1.60^{b}
Monobond Plus (IvoclarVivadent)	9.25±0.86^{b}	10.70±1.71^{b}
Z-PRIME Plus (Bisco Inc.)	10.00±1.70^{b}	14.94±1.70^{c}
CoJet (3M ESPE)	10.38±0.87^{b}	

Y-TZP: yttria-tetragonal zirconia polycrystal.

Values are presented as mean±standard deviation.

Different superscripts indicate a statistical difference ($P<0.05$), and identical superscripts indicate no statistical difference in the designated group after the Student-Newman-Keuls multiple comparison test.

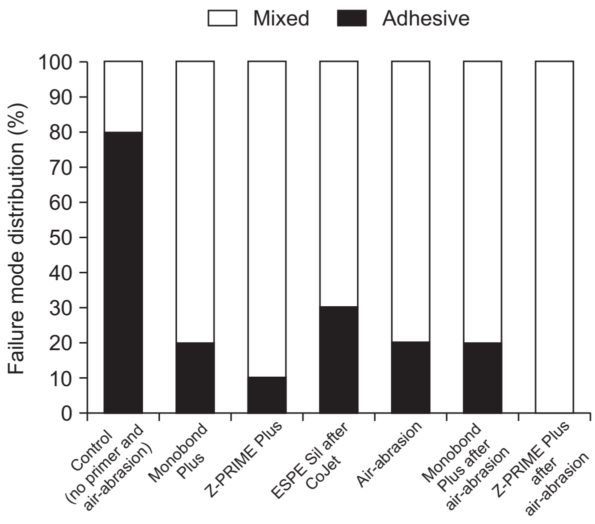

Fig. 2. Failure mode distribution according to the yttria-tetragonal zirconia polycrystal (Y-TZP) surface treatment.

percentage of adhesive failures at the Y-TZP surface in the control group. In contrast, when using additional surface treatments, the mixed failure mode increased.

Fig. 3 shows the representative SEM images (×3,000) of Y-TZP specimens after surface treatment. The polygonal crystals of the Y-TZP can be seen in Fig. 3A, whereas the specimens showed roughened surface after air-abrasion (Fig. 3B). Visible change in Y-TZP surface can be observed after primer application (Fig. 3C, D). The Z-PRIME Plus group (Fig. 3D) showed thicker layer appearance than the Monobond-Plus group (Fig. 3C). A substantial number of silica particles could be seen on the Y-TZP surface after CoJet surface treatment (Fig. 3E)[9]. However, only a small amount of silica could be seen after the water rinsing, which indicates an air-abraded roughened surface structure (Fig. 3F)[9].

Discussion

This study investigated the effect of various surface treatments including the use of conventional methods and the newly developed MDP-containing zirconia primers on the shear bond strength when non-MDP-containing resin cements were used on Y-TZP ceramic.

The hypothesis of the study was rejected since the untreated Y-TZP surfaces showed the lowest bond strength. This result is consistent with the previous studies, which indicated that some conventional resin cements showed low bond strength values when applied to an untreated Y-TZP ceramic surface[9,16,21]. This may be due to the poor chemical interaction between the hydroxyl groups of Y-TZP ceramics and the methacrylate of Variolink II at the interfacial grain level.

The results indicate that not only the conventional methods including air-abrasion and tribochemical silica coating, but also the zirconia primer treatment significantly influenced the bond strength. Air-abrasion with 50 μm-sized particles significantly affected the bond strength regardless of the Y-TZP primer pretreatment. This is consistent with previous studies[9,16,22,23]. The air abrasion method probably increased the roughness and surface energy, thereby facilitating the resin cement flow into microretentions and creating a micromechanical interlocking between the resin cements and Y-TZP. When the tribochemical system is used, silica-modifed Al_2O_3 particles abrade the ceramic surface, and silica particles are included in the ceramic surfaces. After silica-coating, silanization of the silica-modified Y-TZP surface is possible using a silane coupling agent, thus forming a chemical bond with the organic resin materials[24]. During this tribochemical modification, silica is attached to the tough Y-TZP surface, and the underlying mechanismis not completely understood[21]. In order to form a strong and durable bond with Y-TZP, it is extremely important to create a stable and permanent attachment of silica on the Y-TZP surface. However, the bond strength of the tribochemically treated group was comparable to that of the air-abrasion group in the present study. This is probably explained by the fact that the surface roughness of the ceramic was increased by the 30 μm-sized silica particles. These results are

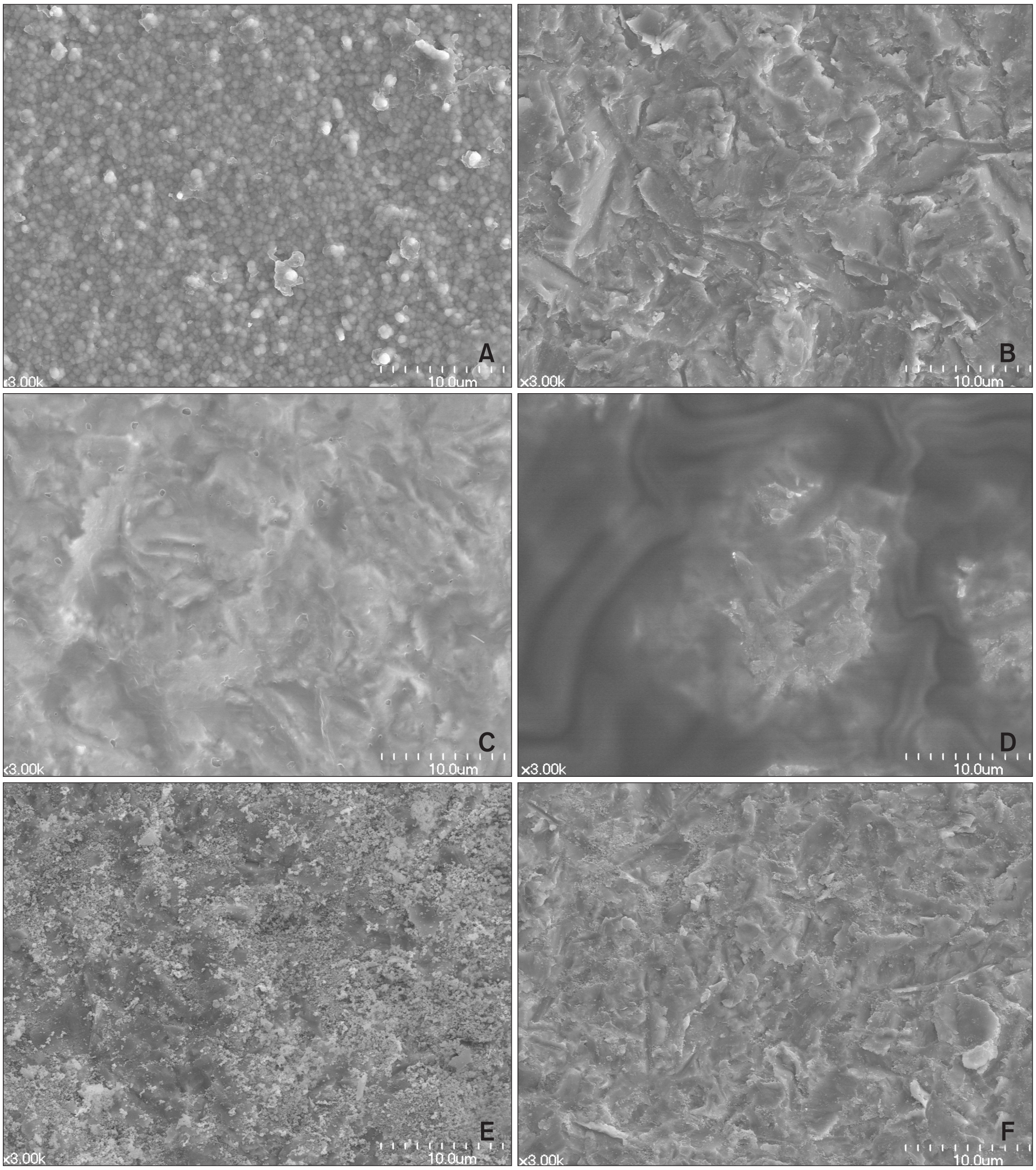

Fig. 3. Scanning electron micrograph images (×3,000) of yttria-tetragonal zirconia polycrystal (Y-TZP) ceramic specimens after surface treatment. (A) The polygonal crystals on an unpolished Y-TZP surface. (B) Roughened surface after air-abrasion with 50 μm grain sized Al_2O_3. (C) Visible changes such as the thin film on the Y-TZP surface were observed after Monobond Plus application (IvoclarVivadent). (D) Visible changes such as the thick film on the Y-TZP surface were observed after Z-PRIME Plus application (Bisco Inc.). (E) According to our previous study, numerous nano-paritcles were present on the Y-TZP surface after the tribochemical CoJet system application (3M ESPE)[9]. (F) Only a small amount of silica could be seen after water rinsing on the CoJet system treated surface[9].

supported by previous studies[9,13]. The SEM images of the abraded zirconia after air-abrasion and tribochemical silica coating both showed increased inter-grain spaces. These enlarged spaces may act as microretentive areas for the resin-based material to flow into and mechanically interlock after polymerization[25]. Moreover, hydroxyl groups that may be generated on the ceramic surfaces during air-abrasion can improve the chemical reaction with MDP[22]. One of the unique properties of Zirconia is the phase transformation from a tetragonal to a monoclinic phase due to the meta-stability[24,26]. While air-abrasion and tribochemical silica coating were significant in increasing the bond strength, they may also induce this phase transformation[9], and it may cause unfavorable changes on the strong mechanical properties of the Y-TZP[24,26].

Treating the Y-TZP surface with Monobond Plus and Z-PRIME Plus significantly increased the shear bond strength. Although the two primers used in the current study contain different compositions, these have MDP monomer in common. Adhesive functional monomers such as MDP do not need to be included in the resin cement if the priming agent contains such monomers. This interpretation is consistent with the results of previous stuies[27,28]. Even though the bond strength is not inferior in non-MDP-containing Variolink II when compared with our previous studies using MDP-containing resin cements applied with the same protocol[9,15], it seems that the MDP in the resin cement doesn't have a synergic effect with the MDP-containing primer. Therefore when MDP-containing primer is applied to the Y-TZP surface, the bond strength is not influenced by whether or not the resin cement contains MDP monomers. MDP included in the primer is a long organic hydrophobic chain molecule with bifunctional ends. Hydrophilic phosphate ester groups on one end, bond strongly to Y-TZP[7,27]. At the other end, vinyl groups react with the monomers of the resin cement during copolymerization[7].

The combination of air-abrasion treatment and Z-PRIME Plus application showed the highest bond strength. It is possible that the improved surface wettability from the air-abrasion treatment and the enhanced chemical affinity from the primer strengthened the bond strength at the bonding interface. In this group, all specimens showed mixed fracture patterns. This was probably because of a synergic effect of increased contact area on the Y-TZP ceramic surface and the increased chemical interaction with the MDP and carboxylic monomer in the Z-PRIME Plus[9]. To form a strong and durable bond to Y-TZP using a non-MDP-containing resin cement, the combination of air-abrasion treatment and MDP-containing primer application is recommended.

Conclusion

Within the limitations of this study, the application of non-MDP-containing resin cement without pretreatment was not sufficient to improve the bond strength to an untreated Y-TZP surface. The combination of air-abrasion treatment and a MDP-containing primer application seems to be a reliable method of forming a strong and durable bond between Y-TZP ceramics and non-MDP-containing resin cements.

Conflict of Interest

No potential conflict of interest relevant to this article was reported.

Acknowledgement

This work was supported by the Basic Science Research Program through the National Research Foundation of Korea (NRF), funded by the Ministry of Education, Science and Technology (2010-

0005090, 2012-009268).

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