Dental zirconia can be etched by hydrofluoric acid

Tool SRIAMPORN1, Niyom THAMRONGANANSKUL2, Chumphol BUSABOK3, Sushit POOLTHONG4, Motohiro UO5 and Junji TAGAMI6,7

1 Faculty of Dentistry, Chulalongkorn University, 34 Henri-Dunant Rd, Patumwan, Bangkok 10330, Thailand
2 Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, 34 Henri-Dunant Rd, Patumwan, Bangkok 10330, Thailand
3 Department of Material Innovation, Thailand Institute of Scientific and Technological Research (TISTR), Pathumthani, 12120, Thailand
4 Department of Operative Dentistry, Faculty of Dentistry, Chulalongkorn University, 34 Henri-Dunant Rd, Patumwan, Bangkok 10330, Thailand
5 Advanced Biomaterials Section, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
6 Cariology and Operative Dentistry, Department of Oral Health Sciences, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
7 Global Center of Excellence Program; International Research Center for Molecular Science in Tooth and Bone Diseases, Graduate School, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan

Corresponding author, Niyom THAMRONGANANSKUL; E-mail: niyom.t@chula.ac.th

The surface morphology and crystal structure change of dental zirconia after hydrofluoric acid (HF) etching were evaluated. Four groups of sintered zirconia specimens were 1) control group, 2) immersion in 9.5%HF at 25°C for 1, 2, 3, or 24 h, 3) immersion in 9.5%HF at 80°C for 1, 3, 5, or 30 min and 4) immersion in 48%HF at 25°C for 30 or 60 min. The specimens were evaluated under SEM and XRD. The SEM analysis revealed changes in surface topography for all the HF-etched zirconia specimens. The irregularities surface increased with increasingly longer immersion times and higher etching solution temperatures. The XRD analysis of the HF-etched zirconia specimens revealed the presence of a crystalline monoclinic phase along with a tetragonal form. It was concluded HF can etch dental zirconia ceramic, creating micro-morphological changes. Tetragonal-to-monoclinic phase transformation was induced on the etched zirconia surface.

Keywords: Acid etching, Hydrofluoric acid, Nano-porosity, Surface treatment, Zirconia

INTRODUCTION

The use of high-strength zirconia ceramics has become increasingly popular in dentistry because of their excellent mechanical properties such as high flexural strength and toughness5-9, esthetic appearance2, and biocompatibility3-5). These ceramics are suitable for a variety of clinical applications, including: posts and cores6,7), dental implants8,9), orthodontic brackets10,11), and fixed-partial dentures12-15). Moreover, zirconia ceramic24) have been developed to improve and enhance the effect of hydrofluoric acid treatment on the surface morphology changes. As a consequence, the aim of this study was to investigate the surface morphology changes. As a consequence, the aim of this study was to investigate the effect of hydrofluoric acid treatment on the surface topography and crystal structure of three commercially available partially-stabilized zirconia ceramics. The
null hypotheses tested were (1) hydrofluoric acid cannot alter the zirconia surface topography, and (2) no crystal structure changes will be present on the etched zirconia surface.

**MATERIALS AND METHODS**

Three commercially available zirconia disk brands were evaluated in this study: KATANA Pre-Sintered Zirconia Block (Kuraray Noritake Dental Inc., Aichi, Japan), Cercon base (DeguDent GmbH, Hanau, Germany), and ZENOTEC Zr Bridge (Wieland, Copenhagen, Denmark). The pre-sintered zirconia disks of each brand were cut into square-shaped specimens (10×10×2 mm) and polished to a flat surface using dry 1200- and 2000-grit silicon carbide abrasive paper and blown with clean dry air for 30 s. The samples were sintered at temperatures recommended in the manufacturers’ instructions. Table 1 shows the brand names, compositions, batch numbers, and manufacturer of the materials used in this study.

Sixty-six specimens prepared from each brand were randomly divided into four groups. For groups 2 and 3, the specimens were divided into four subgroups (n=6) and the group 4 specimens were divided into two subgroups (n=6) according to the surface treatment performed. No treatment was performed for Group 1, which served as the control group.

Group 2: immersion in 9.5% HF at 25°C for 1, 2, 3, or 24 h. Group 3: immersion in 9.5% HF at 80°C for 1, 3, 5, or 30 min. Group 4: immersion in 48% HF at 25°C for 30 or 60 min.

For the immersion procedure, the specimens were immersed in the HF solution contained in a polytetrafluoroethylene beaker and all processing was carried out in a fume hood. After achieving the indicated immersion time, specimens were rinsed with deionized water for 1 min.

Three specimens from each experimental group were randomly selected and ultrasonically cleaned in deionized water for 10 min, and then gently air-dried. The samples were analyzed under a scanning electron microscope (SEM) for surface topography analysis. The three remaining specimens of each of the experimental groups were subjected to X-ray diffraction (XRD) to determine the crystalline composition phases on the zirconia specimen surface from each group. Diffraction patterns were collected within 10–65 2θ at a scan speed of 0.5°/min, covering the area of highest peaks of tetragonal and monoclinic phases of zirconia.

**RESULTS**

SEM images of the zirconia surfaces from each brand are shown in Figs. 1–4. The unetched zirconia surface of the control specimens (Fig. 1) had a homogenous fine grain structure and closed inter-grain space.

Figure 2 shows the zirconia specimen surface from each brand after immersion in 9.5% HF at 25°C for 1, 2, 3, or 24 h. After 1 h of treatment, data regarding the dislodgment of superficial grains, irregular grain structure, decrease in grain size, and enlargement of the inter-grain space compared with the control is shown in Figs. 2a.1, 2b.1, and 2c.1. In addition, with longer treatment times, the images of zirconia HF-treated for 2, 3, or 24 h (Figs. 2a.2–2a.4, 2b.2–2b.4 and 2c.2–2c.4) reveal a further increase in the dislodgment of superficial grains, smaller grains and increased inter-grain space with longer treatment times. Immersion for 24 h resulted in the development of large holes on the zirconia surfaces (Figs. 2a.4, 2b.4 and 2c.4).

The zirconia specimens from each brand immersed in 9.5% HF at 80°C for 1, 3, 5 or 30 min are illustrated in Figs. 3 (3a.1–3a.4, 3b.1–3b.4, and 3c.1–3c.4). The dislodgment of superficial grains, irregular grain shape, decrease in grain size, and enlargement of the inter-grain space was again present as immersion time increased. In addition, large holes were evident after an immersion time of 30 min (Figs. 3a.4, 3b.4 and 3c.4).

| Material | Main composition | Lot number | Manufacturer |
|----------|------------------|------------|--------------|
| KATANA Pre-Sintered Zirconia Block | Zirconium dioxide | KT11BGKLP | Kuraray Noritake Dental Inc., Aichi, Japan |
| Cercon base | Zirconium dioxide | 18010880 | DeguDent GmbH, Hanau, Germany |
| ZENOTEC Zr Bridge | Zirconium dioxide | 6901001140 | Wieland, Copenhagen, Denmark |
| Hydrofluoric acid solution | Hydrofluoric acid in water | B0346834 909 | Merck KGaA, Darmstadt, Germany |
For group 4, images of the zirconia specimens from each brand immersed in 48% HF at 25°C for 30 and 60 min are shown in Figs. 4a.1–4a.2, 4b.1–4b.2 and 4c.1–4c.2. The SEM images reveal irregular morphology similar to that seen in groups 2 and 3. The surface irregularities observed on the specimen surfaces increased with increasing immersion time and higher concentration of the etching solution.

The XRD analysis of all unetched zirconia control surfaces demonstrated a tetragonal phase peak...
Fig. 3  SEM images at 10,000× magnification of zirconia specimen surface from each brand after immersion in 9.5% HF at 80°C for 1, 3, 5 or 30 min, respectively: (a.1–a.4) KATANA Pre-Sintered Zirconia Block, (b.1–b.4) Cercon base and (c.1–c.4) ZENOTECH Zr Bridge.

Fig. 4  SEM images at 10,000× magnification of zirconia specimen surface from each brand after immersion in 48% HF at 25°C for 30 or 60 min, respectively: (a.1–a.2) KATANA Pre-Sintered Zirconia Block, (b.1–b.2) Cercon base and (c.1–c.2) ZENOTECH Zr Bridge.
Fig. 5 XRD analysis of zirconia-oxide ceramic surface, a) control specimen presenting only tetragonal peaks and b) etched zirconia with hydrofluoric acid presenting both tetragonal and monoclinic (*) peaks.

at approximately 30.24 2θ (Fig. 5a). In contrast, a monoclinic phase peak at approximately 28.27 2θ was present in all of the HF treated specimens, as was the tetragonal peak (Fig. 5b).

**DISCUSSION**

The purpose of this study was to evaluate the effect of hydrofluoric acid treatment on the surface microstructure of three dental zirconia ceramics. We found that hydrofluoric acid treatment was able to etch the zirconia ceramic, causing variation in surface topography. In addition, a phase transformation occurred in all the tested groups. Based on these results both null hypotheses were rejected.

Hydrofluoric acid is the inorganic acid hydrogen fluoride. Industrially, hydrofluoritic acid is used to etch silica-based materials, clean metal surfaces before electroplating, and etch silicon wafers in electronic semiconductor materials\(^\text{47-50}\). Concentrations of 4%–10% hydrofluoric acid are typically used in the dental clinic and dental laboratory. These concentration ranges are considered safe for dental applications\(^\text{51}\). Chemically, hydrofluoric acid dissolves glass by reacting with silicon oxide, which is the main ingredient in glass. Dental porcelain consists of glassy and crystal phases, thus HF can dissolve the glassy phase, leaving the crystalline phase, creating surface roughness\(^\text{16,32,33,52-54}\).

We found that immersing zirconia under moderate conditions (9.5% HF at 80°C for 1 min or at 25°C for 1 h) can create surface roughness. We also noted that the irregularities on the specimen surface increased as immersion times were increased and at higher etching solution temperatures. Zirconia treatment with 48%HF at 25°C for 30 min resulted in a highly uneven surface compared with treatment under moderate conditions.

These results are in contrast to previous studies on the etching of zirconia which used concentrations of HF ranging from 4.5–38% with etching times ranging from 1–12 min at room temperature\(^\text{25,36,37,55}\). These studies’ results showed that HF had no effect on the zirconia surface and concluded that HF could not react with the dental zirconia ceramic surface because it is a glass-free material.

We also found that levels of concentration, immersion times, and temperature of the acid solution affected the rate of reaction leading to micro-morphological changes.

The present study showed that hydrofluoric acid can etch the zirconia surface by corroding the zirconia grains. Images of the etched specimen surfaces revealed that the grain size decreased resulting in an increase in the inter-grain space and the zirconia grains were dislodged producing porosities on the surface.

It is known that dental zirconia is a single phase polycrystalline material; therefore, the chemical reactivity depends on the crystallographic orientation. Our study showed the morphology of zirconia surface changed after HF etching. This may be explained in that atoms around the crystal boundaries are more chemically reactive and dissolve faster than those inside the crystal, leading to the formation of irregular grooves around the crystals and grain size was reduction\(^\text{56}\).

Most studies reported that HF treatment did not improve the bond strength between the zirconia surface and resin cement. From our study, HF etched zirconia produced a nano-irregular pattern. This may cause a lower bond strength because the high viscosity the resin cement cannot penetrate into the nano-porosity of the etched-zirconia surface.

Zirconia is a crystalline oxide of zirconium. The crystal structure of pure zirconia can change depending on the temperature: At temperatures below 1,170°C, zirconia exists in the monoclinic form. At temperatures of 1,170°C–2,370°C the monoclinic structure transforms to the tetragonal form, while a cubic structure is present at temperatures over 2,370°C. The addition of metal oxides such as magnesium oxide (MgO), yttrium oxide (Y\(_2\)O\(_3\)), calcium oxide (CaO), and cerium oxide (Ce\(_2\)O\(_3\)) to pure zirconia can inhibit the transformation of the crystalline structure. These oxides can stabilize either the cubic or the tetragonal structure of the material at any temperature. The most effective stabilizing additive is yttrium oxide. Zirconia can be changed into partially stabilized zirconia and fully stabilized zirconia forms. Partially stabilized zirconia, will be in the tetragonal phase at room temperature. Fully stabilized zirconia is required for the cubic phase to be present at room temperature. Partially stabilized zirconia...
can be transformed from tetragonal to monoclinic structure\(^1,4,8,27-65\). This can be caused by pressure or dry conditions known as “transformation toughening”, and moist conditions known as “Low Temperature Degradation (LTD)”\(^61-65\). After etching with hydrofluoric acid under any of the above referenced conditions, all samples in our study exhibited a monoclinic zirconia peak in the XRD analysis. This indicates HF etching can induce the phase transformation of zirconia due to LTD. This likely occurred because the HF etching was applied with prolonged exposure to humidity at low temperature (room temperature –400°C).

While only three brands of dental zirconia ceramic were tested, the findings from this study revealed that HF can etch and corrode the zirconia surface resulting in micro-morphological change and phase transformation. Further investigations are needed to confirm the physical properties in terms of the surface roughness analysis and the surface chemistry of dental zirconia after HF-etching.

CONCLUSION

Within the limitations of our study, it can be concluded that hydrofluoric acid can etch dental zirconia ceramics, creating micro-morphological changes on the zirconia surface. Zirconia surfaces etched with hydrofluoric acid presented the tetragonal-to-monoclinic phase transformation.

ACKNOWLEDGMENTS

This study was financially supported by grants from The 90th Anniversary of Chulalongkorn University Fund (Ratchadaphiseksomphot Endowment Fund) and Faculty of Dentistry, Chulalongkorn University Fund. Prof. John Harcourt, Dr. P.L. Fan, and Dr. Kevin Tompkins are thanked for the proofreading.

REFERENCES

1) Kelly JR, Denry I. Stabilized zirconia as a structural ceramic: an overview. Dent Mater 2008; 24: 289-298.
2) Zarone F, Russo S, Sorrentino R. From porcelain-fused-to-metal to zirconia: clinical and experimental considerations. Dent Mater 2011; 27: 83-96.
3) Uo M, Sjogren G, Sundh A, Watari F, Bergman M, Lerner U. Cytotoxicity and bonding property of dental ceramics. Dent Mater 2003; 19: 487-492.
4) Piconi C, Maccagno G. Zirconia as a ceramic biomaterial. Biomaterials 1999; 20: 1-25.
5) Thompson JY, Stoner BR, Piaskic JR, Smith R. Adhesion/ cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater 2011; 27: 71-82.
6) Akgunor G, Sen D, Aydin M. Influence of different surface treatments on the short-term bond strength and durability between a zirconia post and a composite resin core material. J Prosthodont Dent 2008; 99: 388-399.
7) Ozkurt Z, Iseri U, Kazazoglu E. Zirconia ceramic post systems: a literature review and a case report. Dent Mater J 2010; 29: 233-245.
8) Zinelis S, Thomas A, Syres K, Silikas N, Eliades G. Surface characterization of zirconia dental implants. Dent Mater 2010; 26: 295-305.
9) Denry I, Kelly JR. State of the art of zirconia for dental applications. Dent Mater 2008; 24: 299-307.
10) Keith O, Kusy RP, Whitley JQ. Zirconia brackets: an evaluation of morphology and coefficients of friction. Am J Orthod Dentofacial Orthop 1994; 106: 605-614.
11) Kittipibul P, Godfrey K. In vitro shearing force testing of the Australian zirconia-based ceramic Begg bracket. Am J Orthod Dentofacial Orthop 1995; 108: 308-315.
12) Ardlin BI. Transformation-toughened zirconia for dental inlays, crowns and bridges: chemical stability and effect of low-temperature aging on flexural strength and surface structure. Dent Mater 2002; 18: 590-595.
13) Abou Tara M, Eschbach S, Woffart S, Kern M. Zirconia ceramic inlay-retained fixed dental prostheses —first clinical results with a new design. J Dent 2011; 39: 208-211.
14) Conrad HJ, Seong WJ, Pesun IJ. Current ceramic materials and systems with clinical recommendations: a systematic review. J Prosthet Dent 2007; 98: 388-404.
15) Tauschert J, Natt G, Mohrbritter N, Spiekermann H, Schulze KA. Lifetime of alumina- and zirconia ceramics used for crown and bridge restorations. J Biomed Mater Res B Appl Biomater 2007; 80: 317-321.
16) Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. J Prosthet Dent 2003; 89: 268-274.
17) Yun JY, Ha SR, Lee JB, Kim SH. Effect of sandblasting and various metal primers on the shear bond strength of resin cement to Y-TZP ceramic. Dent Mater 2010; 26: 650-658.
18) Noda M, Okuda Y, Tsuruki J, Minesaki Y, Takenouchi Y, Ban S. Surface damages of zirconia by Nd:YAG dental laser irradiation. Dent Mater J 2010; 29: 536-541.
19) Spohr AM, Borges GA, Junior LH, Mota EG, Oshima HM. Surface modification of In-Ceram Zirconia ceramic by Nd:YAG laser, Rocatec system, or aluminum oxide sandblasting and its bond strength to a resin cement. Photomed Laser Surg 2008; 26: 203-208.
20) Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Selective infiltration-etching technique for a strong and durable bond of resin cements to zirconia-based materials. J Prosthet Dent 2007; 98: 379-388.
21) Casucci A, Osorio E, Osorio R, Montielli F, Toledano M, Mazzitelli C, Ferrari M. Influence of different surface treatments on surface zirconia frameworks. J Dent 2009; 37: 891-897.
22) Casucci A, Mazzitelli C, Montielli F, Toledano M, Osorio R, Osorio E, Papacchini F, Ferrari M. Morphological analysis of three zirconium oxide ceramics: Effect of surface treatments. Dent Mater 2010; 26: 751-760.
23) Jevnikar P, Krnel K, Kocjan A, Funduk N, Kosmac T. The effect of nano-structured alumina coating on resin-bond strength to zirconia ceramics. Dent Mater 2010; 26: 688-696.
24) Phark JH, Duarte S Jr, Blatz M, Sadan A. An in vitro evaluation of the long-term resin bond to a new densely sintered high-purity zirconium-oxide ceramic surface. J Prosthet Dent 2009; 101: 29-38.
25) Qeblawi DM, Munoz CA, Brewer JD, Monaco EA Jr. The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement. J Prosthet Dent 2010; 103: 210-220.
26) Atsu SS, Kilicaslan MA, Kucuksenmen HC, Aka PS. Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. J Prosthet Dent 2006; 95: 430-436.
27) Amaral R, Ozcan M, Bottino MA, Valandro LF. Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: the effect of surface conditioning. Dent Mater 2006; 22: 283-290.
28) Valandro LF, Ozcan M, Amaral R, Leite FP, Bottino MA. Microtensile bond strength of a resin cement to silica-coated
and silanized In-Ceram Zirconia before and after aging. Int J Prosthodont 2007; 20: 70-72.
29) Yoshida K, Yamashita M, Atsuta M. Zirconate coupling agent for bonding resin luting cement to pure zirconium. Am J Dent 2004; 17: 249-252.
30) Yoshida K, Tsuo Y, Atsuta M. Bonding of dual-cured resin cement to zirconia ceramic using phosphate acid ester monomer and zirconate coupler. J Biomed Mater Res B Appl Biomater 2006; 77: 28-33.
31) Kitayama S, Nikaio T, Takahashi R, Zhu L, Ikeda M, Foxton RM, Sadr A, Tagami J. Effect of primer treatment on bonding of resin cements to zirconia ceramic. Dent Mater 2010; 26: 426-432.
32) Canay S, Hersek N, Ertan A. Effect of different acid treatments on a porcelain surface. J Oral Rehabil 2001; 28: 95-101.
33) Borges GA,Sophir AM, de Goes MF, Sobrinho LC, Chan DC. Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics. J Prosthodont 2003; 89: 479-488.
34) Chen JH, Matsumura H, Atsuta M. Effect of etchant, etching period, and silane priming on bond strength to porcelain of composite resin. Oper Dent 1998; 23: 250-257.
35) Yen TW, Blackman RB, Baez RJ. Effect of acid etching on the flexural strength of a feldspathic porcelain and a castable glass ceramic. J Prosthodont 1993; 70: 224-233.
36) Menezes FCH, Borges GA, Valentino TA, Oliveira MAHM, Turussi CF, Correr-sobrinho L. Effect of surface treatment and storage on the bond strength of different ceramic systems. Braz J Oral Sci 2009; 8: 119-123.
37) Derand P, Derand T. Bond strength of luting cements to zirconium oxide ceramics. Int J Prosthodont 2000; 13: 131-135.
38) Della Bona A, Borba M, Benetti P, Cecchetti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. Braz Oral Res 2007; 21: 10-15.
39) Aboushelib MN, Matlininna JP, Salameh Z, Ounsi H. Innovations in bonding to zirconia-based materials: Part I. Dent Mater 2008; 24: 1268-1272.
40) Derand T, Molin M, Kvam K. Bond strength of composite luting cement to zirconia ceramic surfaces. Dent Mater 2005; 21: 1158-1162.
41) Ernst CP, Aksoy E, Stender E, Willershausen B. Influence of different luting concepts on long term retentive strength of zirconia crowns. Am J Dent 2009; 22: 122-128.
42) Janda R, Roulet JF, Wulf M, Tiller HJ. A new adhesive technology for all-ceramics. Dent Mater 2003; 19: 567-573.
43) Magne P, Paranhos MP, Burnett LH Jr. New zirconia primer improves bond strength of resin-based cements. Dent Mater 2010; 26: 345-352.
44) Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Effect of water aging on microtensile bond strength of dual-cured resin cements to pre treated sintered zirconium oxide ceramics. Dent Mater 2009; 25: 392-399.
45) Ozcan M, Nijhuis H, Valandro LF. Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with MDP functional monomer to zirconia after thermal aging. Dent Mater J 2008; 27: 99-104.
46) Donovan TE. Factors essential for successful all-ceramic restorations. J Am Dent Assoc 2008; 139(Suppl4): 14S-18S.
47) Scalzo AJ, Blume-Odom CM. Hydrofluoric acid and other fluorides. In: Shannon MW, Borron SW, Burns MJ, editors. Haddad and Winchester's Clinical Management of Poisoning and Drug Overdose. 4th ed. Philadelphia: Saunders Elsevier; 2007. p. 1323-1334.
48) Genuino HC, Opeomne NN, Njagi EC, McClain S, Suib SL. A review of hydrofluoric acid and its use in the car wash industry. J Ind Eng Chem 2012; 18: 1529-1539.
49) Hatzifotis M, Williams A, Muller M, Pegg S. Hydrofluoric acid burns. Burns 2004; 30: 156-159.
50) Ozcan M, Allahbeikkaraghi A, Dundar M. Possible hazardous effects of hydrofluoric acid and recommendations for treatment approach: a review. Clin Oral Investig 2012; 16: 15-23.
51) Alex G. Preparing porcelain surfaces for optimal bonding. Compend Contin Educ Dent 2008; 29: 324-335.
52) Pisani-Proenca J, Erhardt MC, Valandro LF, Gutierrez-Aceves G, Bolanos-Carmona MV, Del Castillo-Salmeron R, Bottino MA. Influence of ceramic surface conditioning and resin cements on microtensile bond strength to a glass ceramic. J Prosthodont Dent 2006; 96: 412-417.
53) Chen JH, Matsumura H, Atsuta M. Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain. J Dent 1998; 26: 53-58.
54) Thurmond JW, Barkmeier WW, Wilwerding TM. Effect of porcelain surface treatments on bond strengths of composite resin bonded to porcelain. J Prosthodont Dent 1994; 72: 355-359.
55) Komine F, Fushiki R, Koizuka M, Taguchi K, Kamio S, Matsumura H. Effect of surface treatment on bond strength between an indirect composite material and a zirconia framework. J Oral Sci 2012; 54: 39-46.
56) Callister WD Jr, Rethwisch DG, editors. Materials science and engineering: An introduction. 8th ed. New York: John Wiley and Sons, Inc.; 2010.
57) Sundh A, Molin M, Sjogren G. Fracture resistance of yttrium oxide partially-stabilized zirconia all-ceramic bridges after veneering and mechanical fatigue testing. Dent Mater 2005; 21: 476-482.
58) Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part II. Zirconia-based dental ceramics. Dent Mater 2004; 20: 449-456.
59) Tinschert J, Natt G, Mautsch W, Augthun M, Spiekermann RM, Sadr A, Tagami J. Effect of primer treatment on bonding of porcelain surfaces for optimal bonding. J Oral Rehabil 2001; 28: 95-101.
60) Ernst CP, Aksoy E, Stender E, Willershausen B. Influence of different luting concepts on long term retentive strength of zirconia crowns. Am J Dent 2009; 22: 122-128.
61) Janda R, Roulet JF, Wulf M, Tiller HJ. A new adhesive technology for all-ceramics. Dent Mater 2003; 19: 567-573.
62) Magne P, Paranhos MP, Burnett LH Jr. New zirconia primer improves bond strength of resin-based cements. Dent Mater 2010; 26: 345-352.
63) Oyague RC, Monticelli F, Toledano M, Osorio E, Ferrari M, Osorio R. Effect of water aging on microtensile bond strength of dual-cured resin cements to pre-treated sintered zirconium oxide ceramics. Dent Mater 2009; 25: 392-399.
64) Ozcan M, Nijhuis H, Valandro LF. Effect of various surface conditioning methods on the adhesion of dual-cure resin cement with MDP functional monomer to zirconia after thermal aging. Dent Mater J 2008; 27: 99-104.
65) Donovan TE. Factors essential for successful all-ceramic restorations. J Am Dent Assoc 2008; 139(Suppl): 14S-18S.