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Change of phase transformation and bond strength of Y-TZP with various hydrofluoric acid etching

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

Author Contributions
Conceptualization: Lee KW, Yu MK. Data curation: Lim MJ, Oh EJ. Formal analysis: Lim MJ, Oh EJ. Methodology: Yu MK, Oh EJ. Software: Yu MK, Lim MJ. Supervision: Lee KW. Writing - original draft: Lee KW, Yu MK. Writing - review & editing: Lee KW, Lim MJ.

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ABSTRACT

Objectives: The purpose of this study was to quantify phase transformation after hydrofluoric acid (HF) etching at various concentrations on the surface of yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), and to evaluate changes in bonding strength before and after thermal cycling.
Materials and Methods: A group whose Y-TZP surface was treated with tribochemical silica abrasion (TS) was used as the control. Y-TZP specimens from each experimental group were etched with 5%, 10%, 20%, and 40% HF solutions at room temperature for 10 minutes. First, to quantify the phase transformation, Y-TZP specimens ($n = 5$) treated with TS, 5%, 10%, 20% and 40% HF solutions were subjected to X-ray diffraction. Second, to evaluate the change in bond strength before and after thermal cycling, zirconia primer and MDP-containing resin cement were sequentially applied to the Y-TZP specimen. After 5,000 thermal cycles for half of the Y-TZP specimens, shear bond strength was measured for all experimental groups ($n = 10$).
Results: The monoclinic phase content in the 40% HF-treated group was higher than that of the 5%, 10%, and 20% HF-treated groups, but lower than that of TS-treated group ($p < 0.05$). The 40% HF-treated group showed significantly higher bonding strength than the TS, 5%, and 10% HF-treated groups, even after thermal cycling ($p < 0.05$).
Conclusions: Through this experiment, the group treated with SiO_2 containing air-borne abrasion on the Y-TZP surface showed higher phase transformation and higher reduction in bonding strength after thermal cycling compared to the group treated with high concentration HF.

Keywords: Y-TZP; Hydrofluoric acid; Phase transformation; Thermal cycling; Bond strength

INTRODUCTION

The excellent mechanical properties and biocompatibility of yttrium-stabilized tetragonal zirconia (Y-TZP) make it one of the most popular restorative biomaterials. However, Y-TZP has no silica glass component. In addition, since Y-TZP has a homogeneous and densely packaged structure, it is difficult to etch and silanize [1,2]. For this reason, efforts have been made, which are still ongoing, to develop effective surface treatments for Y-TZP to improve its bonding with resin cement.

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A strong and reliable bond between Y-TZP and resin cement relies on both chemical and micro-mechanical bonds [3,4]. According to Nagaoka *et al*. [5], chemical bonding between 10-MDP and Y-TZP is the result of ionic bonding between the dihydrogen phosphate group in 10-MDP and zirconium, and hydrogen bonding between the dihydrogen phosphate group in10-MDP and the hydroxyl group on the surface of Y-TZP.

Various attempts have been made to increase the micro-mechanical retention between Y-TZP and resin cement. Airborne-particle abrasion with Al_2O3 or SiO_2 powder has been evaluated and positive effects reported in several studies [6-8]. In addition, selective infiltration etching and hydrofluoric acid (HF) etching have been proposed to increase bonding strength [9,10]. Until recently, HF etching of Y-TZP was generally considered impossible. However, recent studies have shown that HF etching is possible by adjusting various application conditions, including concentration, immersion time, and temperature [11,12]. According to Liu *et al*. [13], surface treatment using a high concentration HF solution for 25 minutes at 100°C and a silica coating is effective in forming a micro-retentive structure, as well as improving bonding between resin and Y-TZP without phase transformation from a tetragonal to a monoclinic structure on the Y-TZP surface.

The reliability and long-term durability of the bond between Y-TZP and resin are closely related to the life of the restoration itself [14,15]. Resin cement, including MDP, shows excellent adhesion and remains stable even after thermal cycling treatment and long-term storage in water [16,17]. In addition, Lim *et al*. [18] have reported that when a primer containing 10-MDP and resin cement were used in succession, the chemical bond between Y-TZP and resin cement was enhanced, resulting in relatively high bonding strength. However, other studies have reported that although MDP-containing resin cement can improve bond strength through chemical bonding with Y-TZP, bond strength may decrease after thermal cycling treatment [19,20].

The continuous attempts to modify the surface of Y-TZP can greatly contribute to improving the quality of adhesion with MDP-containing resin cement. This study thus aims to quantify phase transformation after HF etching on the Y-TZP surface at various concentrations, and then to evaluate changes in bond strength before and after thermal cycling. The tested null hypothesis is that first of all, there will be no difference in monoclinic phase content even if the Y-TZP surface is subjected to HF etching at various concentrations. Second, even if the Y-TZP surface is subjected to HF etching at various concentrations, the bond strength does not change after thermal cycling.

MATERIALS AND METHODS

Table 1 lists the materials used in this study. The pre-sintered Y-TZP block (NexxZr T, Sagemax, Federal Way, WA, USA) was transformed into standardized cylinders to be used for X-ray diffraction (XRD) analysis and the shear bond strength (SBS) test. Y-TZP cylinders were prepared in 2 different sizes. For XRD analysis, Y-TZP specimens in the form of standard cylinders with diameter of 10 mm and height of 5 mm were prepared. For SBS testing, Y-TZP specimens in the form of standard cylinders with diameter of 12 mm and height of 8 mm were prepared. The Y-TZP specimens were then sintered as follows: 2 to 3 hours at a heating rate of 8°C/min from 0°C to 1,000°C, and 3–4°C/min from 1,000°C to 1,530°C for 3 hours, and finally 2 hours at 1,530°C.

Table 1. Materials used, manufacturers and major components

Material/trade name	Manufacturer	Main components
Y-TZP ceramic/NexxZr T (shade: A1)	Sagemax, Federal Way, WA, USA	91.6% ZrO_2, 5% Y_2O_3, 3% HfO_2
Tribochemical silica/Rocatec Plus	3M ESPE, St.Paul, MN, USA	Aluminum oxide 110 µm modified silica
Hydrofluoric acid/Hydrofluoric acid	Merck KGaA, Darmstadt, Germany	48 wt% in H_2O
Zirconia Primer/Z-prime Plus	Bisco Inc., Schaumbrug, IL, USA	BPDM, HEMA, Ethanol
MDP-containing resin cement/G-CEM LinkAce	GC Corp., Tokyo, Japan	Paste A: Fluoro-alumino-silicate glass, UDMA, Dimethacrylate, Silicon dioxide
		Paste B: Phosphoric acid ester monomer (MDP), Silicon dioxide, UDMA, Dimethacrylate

BPDM, biphenyl dimethacrylate; HEMA, hydroxyethyl methacrylate; UDMA, urethane dimethacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

Specimen preparation

Tribochemical silica air-borne particles (Rocatec plus, 3M ESPE, St. Paul, MN, USA) were used to abrade the surface of the Y-TZP specimens for 10 seconds at a distance of 10 mm at 0.3 MPa. The group whose Y-TZP surface was treated with tribochemical silica abrasion (TS) was used as the control. HF solutions at various concentrations of 5%, 10%, 20%, 40% were prepared from 40% HF solution (MKBH5499V, Sigma-Aldrich Co., St. Louis, MO, USA) using distilled water and an electronic scale. At room temperature, Y-TZP specimens were etched in a plastic box for 10 minutes with 5%, 10%, 20% and 40% HF solutions, then classified into each experimental group. After the HF etching procedure was completed, the Y-TZP specimens were cleaned ultrasonically with distilled water and then gently air-dried.

XRD

To quantify the phase transformation, XRD analysis was performed on each specimen in the tribochemical air-borne abrasion (TA), 5%, 10%, 20% and 40% HF treatment groups ($n = 5$). Prior to this, the XRD patterns of the sintered Y-TZP specimens were obtained and used as a control. Scans were performed at 45kV and 35mA, from 10° and to 80°, with a step size of 0.039°, at 1.31 seconds per step. The XRD patterns were analyzed using Rietveld refinement methods. The monoclinic peak intensity ratio (X_m) was calculated using the equation reported by Garvie and Nicholson [21], as follows:

$$X_m = \frac{I_m(\bar{1}11) + I_m(111)}{I_m(\bar{1}11) + I_m(111) + I_t(111)}$$

where I_t and I_m represent the integrated intensities of tetragonal (111)t peaks and monoclinic (111)m, (-111)m peaks around 2Θ = 30.2°, 31°, and 28.2°, respectively. Monoclinic phase content (F_m) was calculated using the equation reported by Toraya *et al.* [22], as follows:

$$F_m = \frac{1.311 X_m}{1 + 0.311 X_m}$$

SBS

To evaluate the change in bond strength before and after thermal cycling, resin blocks were prepared by incrementally packing a flowable resin (Unifil Loflo, GC Inc., Tokyo, Japan) into a cylindrical Teflon mold (4 mm diameter, 8 mm height) then light-cure for 20 seconds at each increment. Zirconia primer (Z-prime plus, Bisco Inc., Schaumburg, IL, USA) was first applied to the Y-TZP surface for 10 seconds and air-dried for 15 seconds in all experimental groups ($n = 10$). Then, for cementation, resin cement containing MDP (G-CEM LinkAce, GC Inc.) was placed between the Y-TZP specimen and the resin block. Light curing was performed for 3 seconds for tack curing under a constant load of 5N and excess cement was carefully removed using a dental explorer. While maintaining constant load, light irradiation

was added to the adhesive surface for 20 seconds. After the luting procedure was completed, all Y-TZP specimens were stored in distilled water at 37°C for 24 hours. Half of the Y-TZP specimens were then additionally thermocycled at 5,000 cycles between 5°C and 55°C with a dwell time of 20 seconds prior to the SBS test. The SBS test was conducted with a universal testing machine (Model 5543, Instron, Canton, MA, USA) and shear force (using a chisel-shaped metal rod at a constant crosshead speed of 0.5 mm/min) was loaded to the specimens until the resin block column was dropped. The maximum load (recorded in N) was measured by recording the force at which failure occurred, and the bond strength of each specimen was calculated in MPa by dividing the maximum load (N) by the bonding surface area (mm^2).

Scanning electron microscopy (SEM)
After SBS measurement, the debonded surfaces were analyzed using SEM (JSM-6400, JEOL Co., Tokyo, Japan) to determine the failure mode between the Y-TZP and resin cement.

Statistical analysis
Statistical analysis was performed using statistical software (SPSS Version 18.0, SPSS Inc., Chicago, IL, USA). Mean and standard deviation were statistically analyzed using one-way analysis of variance. Statistical significance between the experimental groups for the monoclinic phase content (F_m.%) calculated by XRD analysis was verified using the Mann Whitney test, and significant differences of SBS values between groups were determined using the pairwise multiple comparison procedure (Tukey's test). The statistical significance of the rate of decrease in bond strength between experimental groups at each of the same HF concentrations was analyzed by t-test. A p value less than 0.05 was considered statistically significant in all tests.

RESULTS

XRD analysis
The XRD patterns of the Y-TZP surface treated with tribochemical air-borne abrasion (TA) and 5%, 10%, 20%, and 40% HF are shown in **Figure 1**. The monoclinic phase content (F_m.%) of each experimental group is shown in **Figure 2**, and the mean ± standard deviation of each TA, 5%, 10%, 20%, 40% HF treatment groups was as follows; 7.78 ± 1.07, 1.89 ± 0.05, 1.55 ± 0.05, 1.97 ± 0.1, 2.99 ± 0.07, respectively. The analysis results revealed that monoclinic phase content (F_m.%) was significantly higher in the TA group than in the other experimental groups ($p < 0.05$). Among the 5%, 10%, and 20% HF treatment groups, the ratio of phase transformation to increased concentration was not statistically significant ($p > 0.05$). In the group treated with 40% HF, monoclinic phase content was higher than in the 5%, 10%, and 20% HF treatment groups, but remained lower than the TA group ($p < 0.05$).

SBS
Figure 3 shows the bond strength between the Y-TZP surface and the resin cement before and after thermal cycling in tribochemical air-borne abrasion (TA) and 5%, 10%, 20% and 40% HF treatment groups. The experimental group etched with 40% HF before thermal cycling showed significantly higher bond strength than the TA, 5% and 10% HF treated groups ($p < 0.05$). Even after thermal cycling, the group treated with 40% HF showed significantly higher adhesion than the groups treated with TA, 5% and 10% HF ($p < 0.05$). **Table 2** shows the rate of decrease in bonding strength after thermal cycling in the TA group and the HF treatment group at the same concentration. It was observed that bond strength decreased
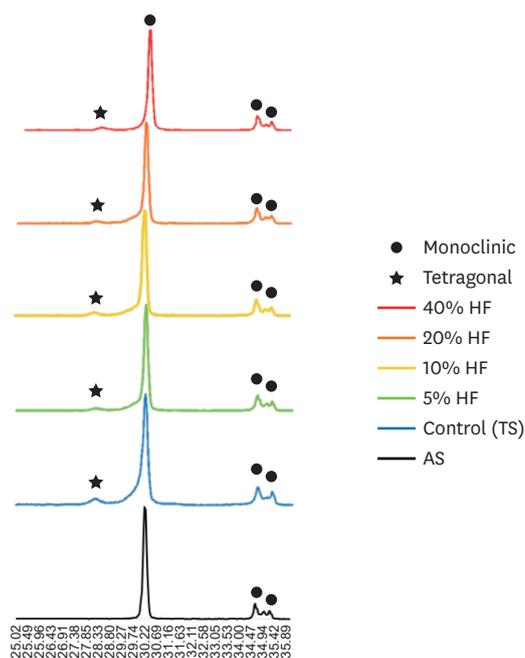
Figure 1. X-ray diffraction patterns of yttria-stabilized tetragonal zirconia polycrystal surfaces etched with various concentrations of hydrofluoric acid or treated with tribochemical silica abrasion.
TS, tribochemical silica air-borne abrasion; AS, after sintering.
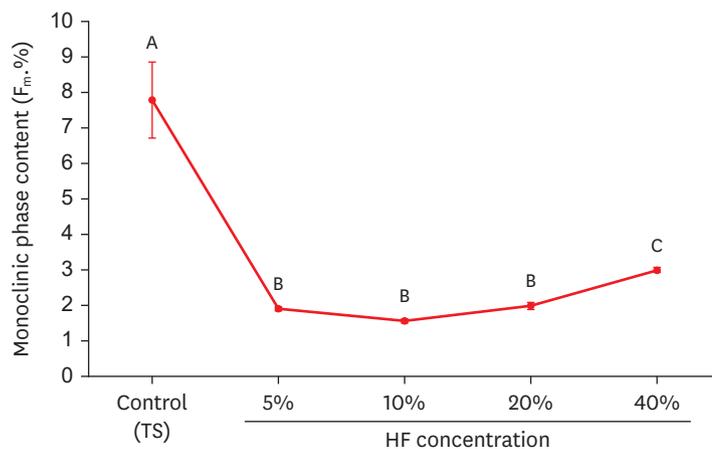
Figure 2. Monoclinic phase content ($F_m.\%$) of yttria-stabilized tetragonal zirconia polycrystal when treated with hydrofluoric acid depending on concentration conditions. Identical capital letters in figure indicate no statistically significant differences ($p > 0.05$).
TS, tribochemical silica air-borne abrasion; HF, hydrofluoric acid.

Table 2. Rate of decrease in bond strength after thermal cycling in experimental group for hydrofluoric acid etched yttria-stabilized tetragonal zirconia polycrystal under various concentration conditions

Concentration	0% (TS*)	5%	10%	20%	40%
SBS before TC (MPa)	17.40	16.73	20.23	21.61	26.64
SBS after TC (MPa)	8.37	11.51	10.77	14.39	17.10
Strength reduction rate (%)	51.9[†]	31.2	46.7[†]	33.4[†]	35.8[†]

SBS, shear bond strength; TC, thermal cycling.
*TS, tribochemical silica air-borne abrasion; [†]indicates statistically significant differences ($p < 0.05$).
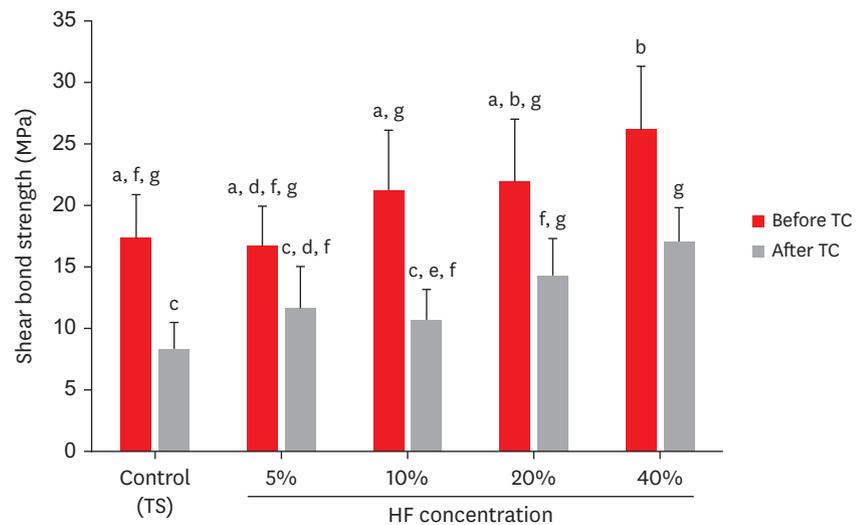
Figure 3. Shear bond strength before and after thermal cycling for HF etched yttria-stabilized tetragonal zirconia polycrystal at various concentration conditions. Identical lowercase letters indicate no statistically significant differences ($p > 0.05$).
TS, tribochemical silica air-borne abrasion; TC, thermal cycling; HF, hydrofluoric acid.

significantly after thermal cycling among experimental groups of the same concentration, except in the group treated with 5% HF ($p < 0.05$). In particular, the bonding strength was most significantly decreased in the control TA group ($p < 0.05$).

SEM observation after debonding

Figure 4 shows SEM of the debonded Y-TZP specimens after measurement of bond strength without thermal cycling. Specimens treated with 20% and 40% HF concentrations (**Figure 4D and 4E**) showed predominantly mixed failure modes, whereas specimens treated with TA, 5% and 10% HF (**Figure 4A, 4B, and 4C**) showed mainly adhesive failure. The primed layer could be clearly observed at high magnification in the HF treatment groups with a concentration of 10% or more (**Figure 4H, 4I, and 4J**). **Figure 5** shows the SEMs of the debonded Y-TZP specimens after measurement of bond strength after thermal cycling. Even after this, the specimens treated with 20% and 40% HF (**Figure 5D and 5E**) showed mixed failure mode,
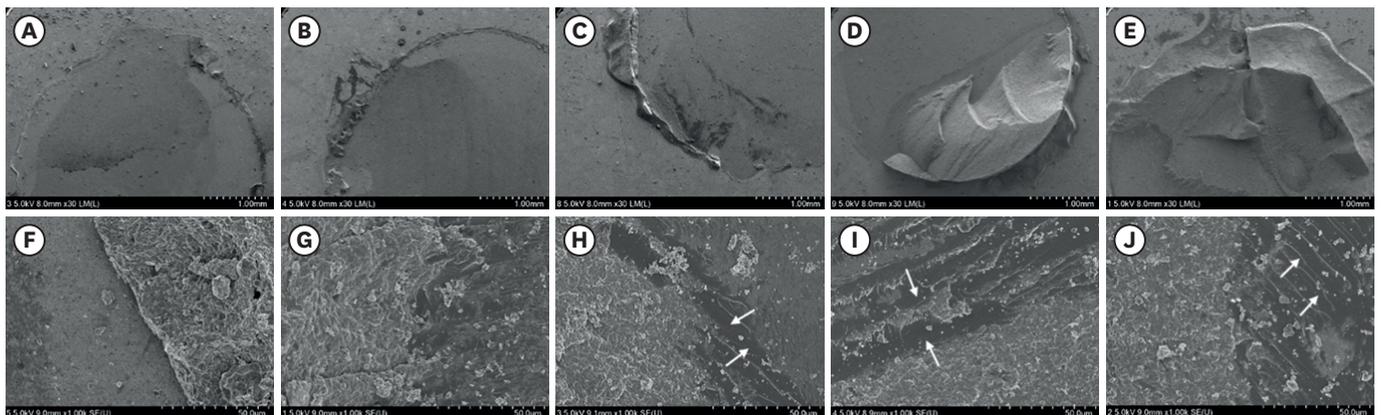
Figure 4. Scanning electron microscopic views of debonded Y-TZP specimens without thermal cycling after surface treatment with various concentrations of hydrofluoric acid. (A-E) Y-TZP specimens treated with tribochemical silica abrasion, 5%, 10%, 20%, and 40% HF at magnification X30. (F, J) Y-TZP specimens treated with tribochemical silica abrasion, 5%, 10%, 20%, and 40% HF at magnification ×10,000. The white arrow indicates the primed layer.
Y-TZP, yttria-stabilized tetragonal zirconia polycrystal.
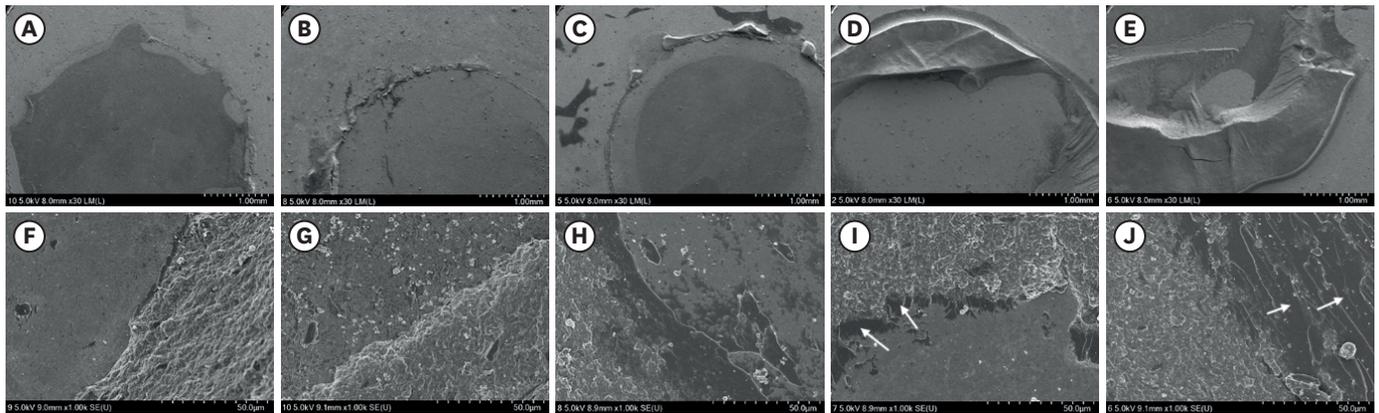
Figure 5. Scanning electron microscopic views of debonded Y-TZP specimens thermally cycled after surface treatment with various concentrations of hydrofluoric acid. (A-E) Y-TZP specimens treated with tribochemical silica abrasion, 5%, 10%, 20%, and 40% HF at magnification X30. (F, J) Y-TZP specimens treated with tribochemical silica abrasion, 5%, 10%, 20%, and 40% HF at magnification ×10,000. The white arrow indicates the primed layer.
Y-TZP, yttria-stabilized tetragonal zirconia polycrystal.

while the specimens treated with TA and lower HF concentrations (**Figure 5A, 5B, and 5C**) showed adhesion failure mode. However, at high magnification, primed layers were observed only in the specimens treated with HF concentrations of 20% or more (**Figure 5I and 5J**).

DISCUSSION

There have been studies of an air-borne abrasion method that increases the roughness of the Y-TZP surface and induces high bonding strength by using a phosphate ester monomer [13,23]. This removes impurities, induces surface modification, and increases the surface roughness (Ra) of Y-TZP to improve micro-mechanical retention. However, air-borne abrasion can be affected by variables such as particle size, injection distance, time, and pressure [24,25]. In addition, this method also induces phase transformation of the Y-TZP surface crystal structure from tetragonal to monoclinic. The formation of microcracks from these deformations can negatively affect the long-term stability of Y-TZP [26,27]. In the present study, air-borne abrasion containing SiO_2 with a particle size of 110 μm was selected instead of air-borne abrasion using Al2O3. Similar to the report of Kang *et al.* [28] using Al_2O_3, in this experiment, the experimental group treated with air-borne abrasion containing SiO_2 showed significantly higher phase transformation than all the experimental groups treated with HF.

In this study, phase transformation occurred in the experimental group whose Y-TZP surface was treated with air-borne abrasion containing SiO_2, as well as in all experimental groups treated with various concentrations of HF (**Figures 1** and **2**). This shows that HF etching can also cause phase transformation on the Y-TZP surface [29,30]. However, although the tetragonal to monoclinic phase transformation was observed in all HF-treated groups, the monoclinic phase content of all HF-treated groups was smaller than that of the air-borne abrasion group, which was insufficient to adversely affect bonding strength.

Although HF is the most commonly used agent for etching dental ceramics, it was reported that the Y-TZP surface did not change even when treated with HF solution because it was a glass-free material [1,3]. However, in several recent studies treating Y-TZP surfaces with HF solutions of various concentrations and temperatures, it was reported that increased surface roughness and porosity could improve the bonding strength [27,31]. Also, Kim *et*

al. [12] reported that the surface roughness of Y-TZP can increase with the change of HF concentration and temperature, which has a positive effect on the bonding conditions.

In this study, the effect of HF etching of various concentrations on the adhesive strength between Y-TZP and resin cement after thermal cycling was investigated. The results showed that bonding strength after thermal cycling decreased significantly both in the air-borne abrasion group containing SiO_2 and the HF treatment groups (**Figure 3**). This is because, as Attia [32] has argued, the improved bonding strength in Y-TZP cannot be maintained for a long time due to the acceleration of hydrolysis at the bonding site. However, in this experiment, the experimental group etched with 40% HF even after thermal cycling showed significantly higher adhesion than the tribochemical air-abrasion group and the 5% and 10% HF treatment groups (**Figure 3**). This is believed to be because the Y-TZP surface is changed to a uniform nanostructure by the high concentration of the HF treatment, and the resin cement is easily wetted and penetrates into this structure, forming a micromechanical bond. It can also be assumed that bonding strength is further enhanced by the chemical bond between Y-TZP and MDP-containing resin cement.

In this experiment to improve the bonding strength between Y-TZP and MDP-containing resin cement, the group treated with air-borne abrasion containing SiO_2 showed a greater degree of phase transformation than the group treated with high concentration of HF (**Figure 2**). In addition, although a reduction in bonding strength was observed in all experimental groups after thermal cycling, it was confirmed that the reduction rate of the SiO_2-containing air-borne abrasion group was significantly greater than for the HF-treated group (**Table 2**). Based on the results of this study, therefore, each of the null hypotheses initially proposed was rejected.

Through this experiment, it was confirmed that if the Y-TZP surface was treated with a high concentration of HF at room temperature for 10 minutes, its bonding strength with the resin cement increased and could be maintained even after thermal cycling. However, Ding *et al.* [33] have suggested that although HF application may improve bonding strength, especially at high concentrations it may also reduce fracture resistance, resulting in a problem in durability. It is therefore to be expected that further research will be needed on this issue in the future.

CONCLUSIONS

Within the limits of this experiment, the group treated with SiO_2 containing air-borne abrasion on the Y-TZP surface showed both a higher degree of phase transformation and a higher rate of decrease in bonding strength after thermal cycling compared to the group treated with high concentration HF. It is therefore believed that high concentration HF treatment of the Y-TZP surface may provide an alternative method of achieving both reliability and long-term durability in the bond between Y-TZP and MDP-containing resin cement.

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