In vitro and clinical studies on enamel wear in relation to aging of yttria-partially stabilized zirconias

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감사의 글

본 논문을 완성하는데 있어 아낌없는 격려와 세심한 지도로 저를 이끌어 주신 김지환 지도교수님께 마음 깊이 감사드립니다. 바쁘신 와중에도 논문 심사를 맡아 주시고 바른 방향으로 논문이 작성될 수 있도록 귀중한 조언을 해 주신 심준성 교수님, 김종은 교수님, 신유석 교수님 그리고 권재성 교수님께 깊은 감사를 드립니다. 또한, 치과의사로서 올곧게 성장할 수 있도록 늘 관심으로 지켜봐 주시고 가르침을 주신 정문규 교수님, 한동후 교수님, 이근우 교수님, 문홍석 교수님, 김선재 교수님, 박영범 교수님, 이재훈 교수님, 박지만 교수님께 감사드립니다. 함께 의국 생활을 했던 보철과 동기 및 선후배 선생님들과, 실험에 도움을 주신 이채은 선생님에게도 감사의 마음을 전합니다. 앞으로도 겸손한 자세로 탐구하며 끊임없이 정진하는 치과의사가 되도록 노력하겠습니다.

언제나 제 곁에서 지켜봐 주시며, 물심양면으로 도움을 주시는 아버지, 어머니, 장인어른, 장모님께도 사랑한다는 말씀과 함께 가슴 깊이 감사드립니다. 그리고 언제나 옆에서 저에게 힘이 되어주고, 저를 응원해주는 아내와 이 기쁨을 함께 나누고 싶습니다.

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양 승 원

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ABSTRACT

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Yttria-partially stabilized zirconia (Y-PSZ) with its improved translucency is a widely used dental restorative material. However, enamel wear and phase transformation upon actual application of Y-PSZ has not yet been clarified. This study aimed to evaluate wear of Y-PSZ and antagonistic enamel. Also, surface roughness and aging associated with the use of polished Y-PSZ were evaluated in both in vitro and clinical experiments. The null hypothesis was that wear, surface roughness, and phase transformation would be similar for the two materials and between the in vitro and clinical experiments.

In vitro experiments were performed with Katana (Katana ML Block, Kuraray Noritake Dental Inc., Aichi, Japan) zirconia blocks, Rainbow (Rainbow Shade Block, Genoss Co., Suwon, Korea) zirconia blocks, and natural tooth enamel (control), which were subjected to 100,000 cycles of cyclic loading with a maxillary premolar antagonist. All specimens were scanned for wear analysis, and the zirconia specimens were evaluated for surface roughness and monoclinic phase (m-phase) transformation by X-ray diffractometry (XRD) before and after cyclic loading.

The clinical study included patients who required single-crown restoration on first or second molar implants. The patients received Katana or Rainbow zirconia prostheses (n = 15, each). For wear analysis, impressions of each prosthesis, antagonist, and adjacent tooth were taken at 1 week and 6 months after crown delivery. The ratio of relative strength of prostheses in maximum intercuspation was evaluated using the occlusal diagnostic system (T-Scan 8, Tekscan Inc., South Boston, MA, USA). The degree of transformation of zirconia to the m-phase was measured by XRD of prostheses after 6 months of use.

Overall, zirconia induced significantly greater enamel wear than natural teeth did. Katana specimens exhibited significantly greater wear and surface roughness than Rainbow specimens. The degrees of antagonistic wear and zirconia phase transformation in clinical studies were significantly greater than those in in vitro experiments. Katana groups showed significantly higher m-phase levels than the Rainbow groups did.

Thus, phase transformation of zirconia occurred by clinical application, and the wear and degrees of phase transformation varied according to the difference of zirconia.

Keywords: Aging; Clinical study; Phase transformation; Surface roughness; Wear; Zirconia

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I. INTRODUCTION

Since its introduction in dentistry, zirconia has been widely used for restoration of implant-supported fixed dental prostheses (ISFDP) and natural teeth (Koutayas et al. 2009; Zarone et al. 2011; Stober et al. 2014). Traditional 3 mol.% yttria-stabilized tetragonal zirconia polycrystal (3Y-TZP) had a disadvantage of low esthetic quality because of its opaqueness, which was resolved by the technique of porcelain veneering on zirconia core

(Bachhav and Aras 2011). However, veneered porcelain is continuously subjected to chipping and delamination (Zarone et al. 2011). These problems have been overcome by the use of translucency increased monolithic zirconia.

Decreasing the Al_2O_3 content in Y-TZP reduces light scattering and improves translucency. Also, controlling the yttria content changes the translucency of Y-TZP (Zhang 2014; Zhang et al. 2015). 3Y-TZP has single transformable tetragonal phase. However, as the yttria content increases, 3Y-TZP becomes yttria-partially stabilized zirconia (Y-PSZ) such as 4Y-, 5Y-PSZ with increased cubic phase and translucency. Especially when more than 8 mol.% of yttria is added, fully stabilized zirconia with pure cubic form is produced (Vagkopoulou et al. 2009; Lughi and Sergo 2010). However, wear of antagonistic natural tooth enamel and low-temperature degradation (LTD) of zirconia are complicated.

Wear is a complex phenomenon involving physical, chemical, and biological factors (Ekfeldt and Oilo 1990; Sulong and Aziz 1990; Mair et al. 1996; Yip et al. 2004). It is, therefore, challenging to reproduce complex oral conditions in vitro (Sakaguchi et al. 1986; Heintze 2006). Excessive wear of teeth and restorative materials can lead to instability of occlusion, decrease in vertical dimension and masticatory function, esthetic problems, and incongruity of the oral maxillofacial system (Yip et al. 2004; Mundhe et al. 2015). Therefore, it is important to choose restorative materials that can harmonize with natural tooth wear, on the basis of clinical considerations (Yip et al. 2004).

Depending on the type of prosthesis, the wear of opposing enamel varies. Many studies have reported that, relative to porcelain, zirconia induces lesser wear on antagonistic enamel of natural teeth (Jung et al. 2010; Kim et al. 2012; Stawarczyk et al. 2013; Burgess et al. 2014; Park et al. 2014; Sripetchdanond and Leevailoj 2014; Stober et al. 2014; Mundhe et al. 2015). Polished zirconia has been shown to induce lesser wear of natural enamel than veneered or glazed zirconia (Heintze et al. 2008; Janyavula et al. 2013; Stawarczyk et al. 2013). However, most of these studies on wear analysis have been limited to in vitro experiments.

LTD of zirconia further increases the complexity of the wear process (Passos et al. 2014). Y-TZP maintains a tetragonal phase at room temperature (Piconi and Maccauro 1999; Fabris et al. 2002). However, upon application of external force, crystal nuclei are formed around the point of loading, causing the stabilized tetragonal phase to shift to the monoclinic phase (m-phase). This process is accompanied by a 3–5% volume expansion, which stops the progression of crack lines because of compressive stress, in a phenomenon termed transformation toughening (Hannink et al. 2000). However, in LTD, Y-TZP undergoes spontaneous tetragonal–monoclinic phase transformation on the surface because of the presence of moisture and pressure in the oral cavity, so called aging of zirconia, leading to surface roughness and decreased strength (Chevalier et al. 1999; Kohorst et al. 2012). Whether LTD caused strength degradation is controversial and some studies show the opposite (Pereira et al. 2016; Ramos et al. 2016). Opinions on the effect of strength of zirconia on wear of opposing enamel also vary. Previously, stronger materials were deemed to cause greater wear of natural enamel (Palmer et al. 1991; Oh et al. 2002). However, recent studies have shown that surface roughness of restorative materials, rather than their strength, has a greater effect on wear of opposing enamel (Oh et al. 2002; Heintze et al. 2008; Passos et al. 2014).

In Y-PSZ, aging can affect the wear of opposing natural enamel because it results in decreased strength and increased surface roughness because of surface degradation (Passos et al. 2014). However, few clinical studies have evaluated the effect of zirconia on enamel wear, and no study to date has analyzed aging upon clinical application of zirconia (Stober et al. 2014; Mundhe et al. 2015). Research on phase transformation upon application of zirconia in the oral cavity is necessary (Al-Amleh et al. 2010). Moreover, there is a lack of experimental and clinical data on translucency increased zirconia (Flinn et al. 2017). Further research is also required on the changes in the nature of translucency increased zirconia, including its physical and phase transformation properties.

This study aimed to evaluate wear of Y-PSZ and antagonistic enamel. Also, surface roughness and aging according to the use of polished Y-PSZ were evaluated in both in vitro and clinical experiments. Phase transformation of the clinically used Y-PSZ was evaluated particularly. The null hypothesis was that wear, surface roughness, and phase transformation would be similar for the two materials and between the in vitro and clinical experiments.

II. MATERIALS AND METHODS

1. Two zirconia materials

For the in vitro and clinical experiments, Katana (Katana ML Block, Kuraray Noritake Dental Inc., Aichi, Japan) and Rainbow (Rainbow Shade Block, Genoss Co., Suwon, Korea) zirconia blocks were used. Katana and Rainbow zirconia blocks are zirconias with increased translucency. The composition of each material is detailed in Table 1 (Kim and Kim 2016). Katana has about 7.14 wt.% yttria, which is equivalent to 5.08 mol.% Y-PSZ and Rainbow has about 5.00 wt.% yttria, which is equivalent to 3.56 mol.% Y-PSZ. The higher the yttria content of zirconia, the higher the cubic phase and translucency (Zhang 2014; Zhang et al. 2015).

Table 1. Basic composition of the experimental materials.

Material	Manufacturer	Lot Number.	Composition (wt.%)
Katana ML Block	Kuraray Noritake	DIBOT	ZrO_2, Y_2O_3 7.12–7.16%
Rainbow Shade Block	Genoss	15J05-04	ZrO_2, Y_2O_3 4–6%, $HfO_2 \leq$ 5%, $Al_2O_3 \leq$ 1%

2. In vitro experiments

An overview of the in vitro experiments is shown in Figure 1.

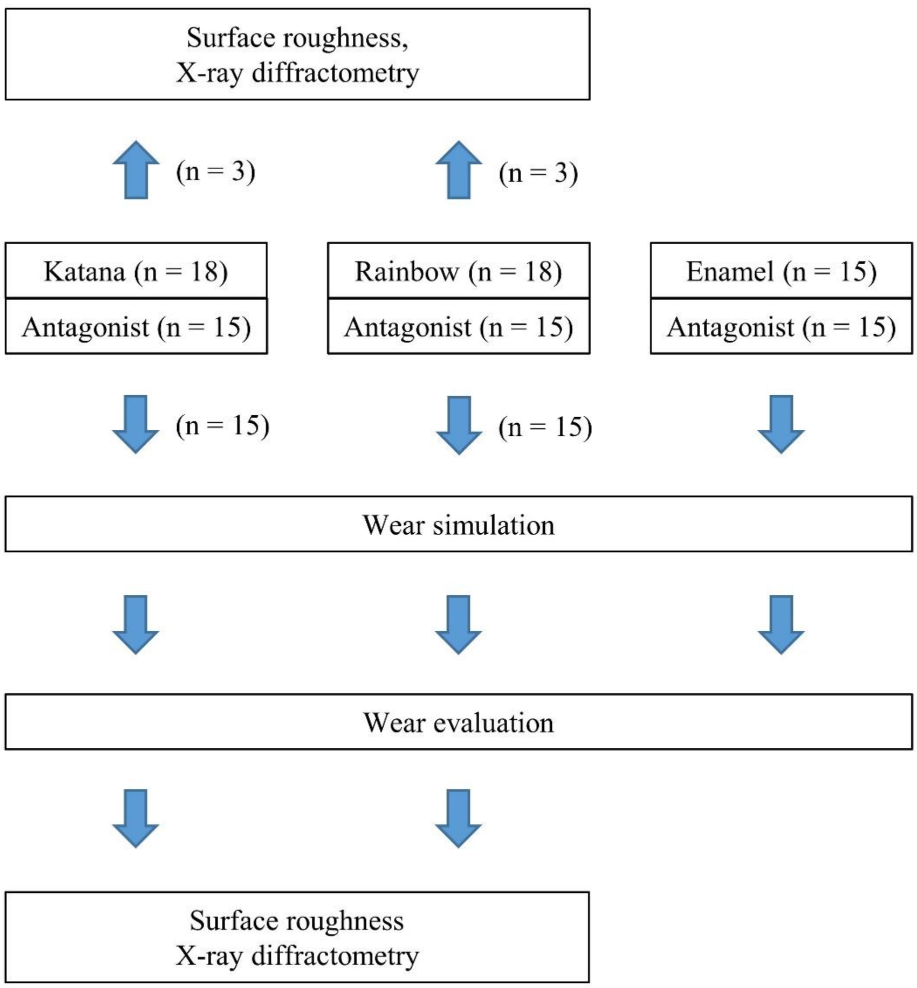

Figure 1. Schematic illustration of in vitro experiments.

2.1. Specimen preparation for experiments

2.1.1. Substrate specimens

For the in vitro experiments, three types of substrate, including Katana (n = 18) and Rainbow (n = 18) zirconia blocks and natural enamel (n = 15), were tested. Zirconia specimens were produced in a rectangular parallelepiped of 7 x 7 x 13 mm size (width x length x height). After sintering, the specimens were polished using zirconia polishing burs (Zirco master, Seichong Co., Seoul, Korea), in accordance with the manufacturer's instructions. Then, Katana and Rainbow specimens were embedded in a specialized jig using acrylic resin (Figure 2A; n = 15, each). Enamel specimens were derived from extracted human maxillary premolars. Premolars with carious lesions or enamel fractures were excluded. Dental roots were removed, and, after ultrasonic cleaning, the crown was polished with pumice powder for uniformity (Janyavula et al. 2013). The flat part of the crown enamel was embedded in a specialized jig using acrylic resin, to make contact with the antagonist.

2.1.2. Antagonist specimens

Extracted human maxillary premolars were used as antagonists. Premolars with fractured, worn, or highly sharp cusps were excluded. After ultrasonic cleaning, the premolars were polished with pumice powder, homogenized (Janyavula et al. 2013), and embedded in a jig using acrylic resin to ensure functional cusp contact with the substrate (Figure 2B).

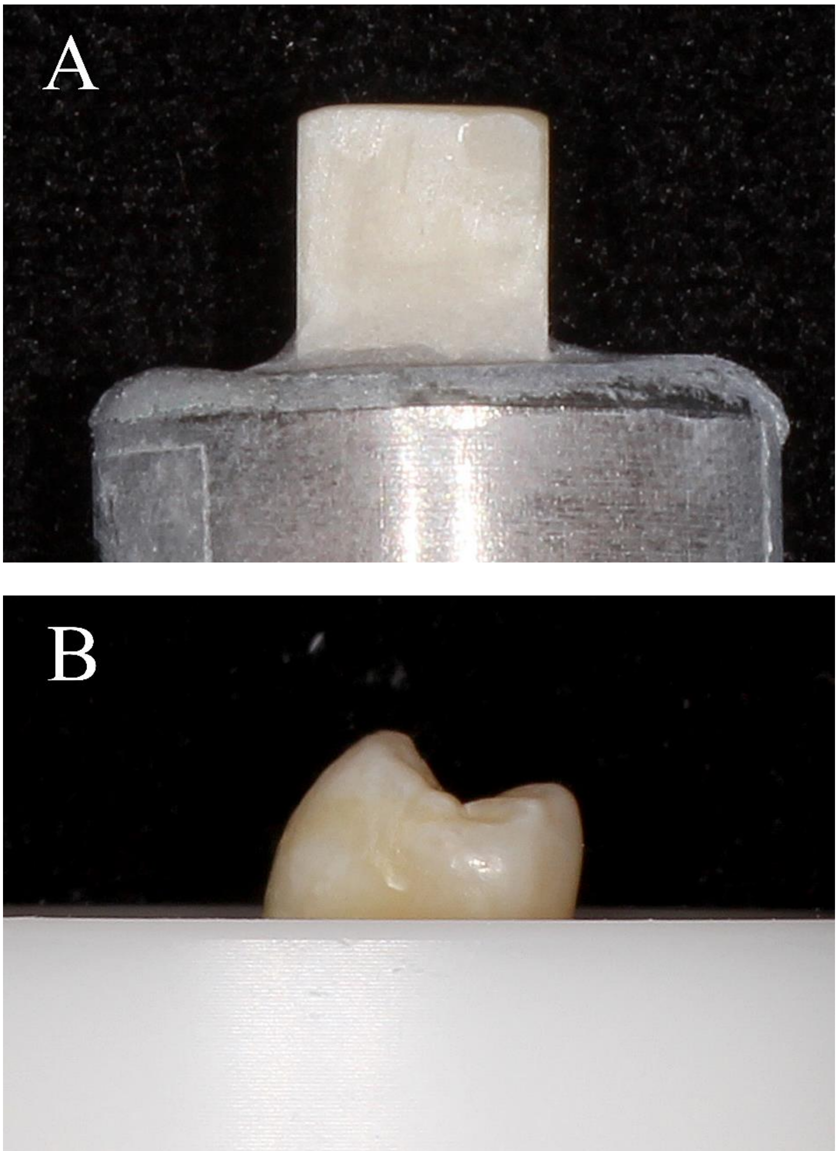

Figure 2. In vitro specimen for wear analysis. (A) Zirconia substrate, (B) Antagonist made from human maxillary premolar.

2.2. Wear simulation

Wear analysis was performed using a chewing simulator (CS-4.8, SD Mechatronics, Feldkirchen-Westerham, Germany). Jigs with substrate and antagonist (15 specimens per group) were mounted on upper and lower holders, respectively (Figure 3). Chewing simulation was performed under conditions of 2-mm vertical and 2-mm horizontal movement, 1.5-hz frequency, and a load of 5 kg, corresponding to a masticating pressure of 49 N (Kim et al. 2012; Janyavula et al. 2013). Then, 100,000 cycles of pressure, corresponding to 6 months of work, were applied (Stober et al. 2006). Each chamber was subjected to heat circulation at 5–55 ℃ (Moore RJ et al. 1999). Cold and hot bath temperatures were set at 5 ℃ and 55 ℃, respectively. Each dwell time was set to 60 seconds, and a total 1250 cycles were performed.

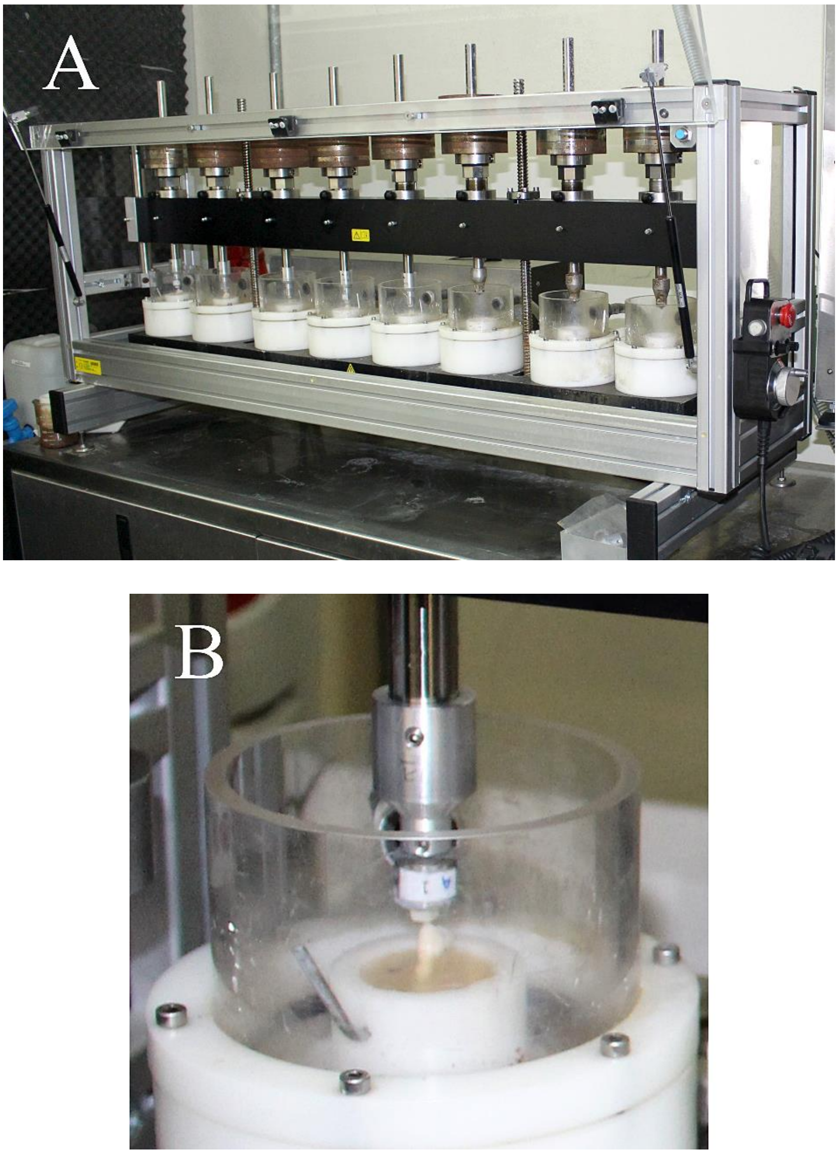

Figure 3. Chewing simulator. (A) CS-4.8 (SD Mechatronics, Feldkirchen-Westerham, Germany). (B) The substrates were mounted on the upper holder and the antagonists on the lower holder using a screw.

2.3. Wear analysis

Fabricated substrates and antagonists were scanned using a blue light-emitting diode, 50 ANSI-lumens desktop scanner (Identica Hybrid, Medit, Seoul, Korea) with a resolution of mono 1.3 mega pixel, color 5.0 mega pixel camera and an accuracy of 7 μm (ISO 12836) before and after wear analysis to generate stereolithography (STL) files. Using a 3D scanner software (Rapidform 2006, INUS Technology and Rapidform Inc., Seoul, Korea), STL files before and after wear analysis were superimposed. The reference points of superimposition were side aspects which were less affected by wear such as buccal and lingual surfaces. The mean and maximum vertical differences caused by the wear test were calculated (Figure 4).

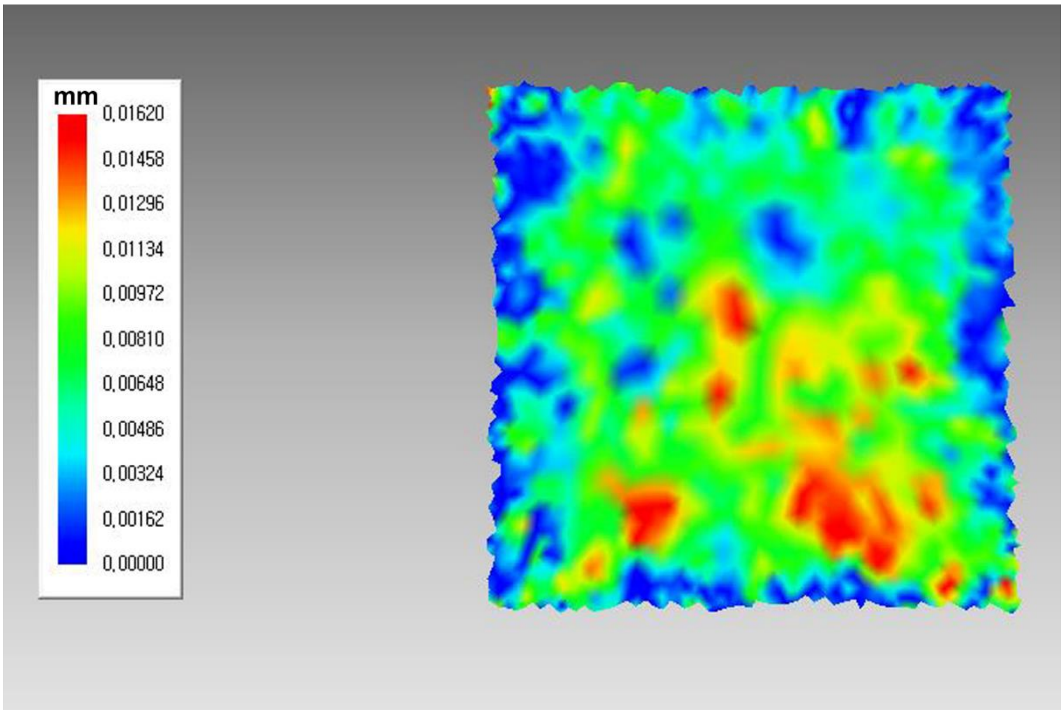

Figure 4. Representative image of in vitro zirconia substrate wear analysis. Red areas mean severe wear after 6 months of function.

2.4. Quantitative analysis of surface roughness and phase transformation before and after wear analysis

Before wear simulation, Surface roughness was measured using a three-dimensional (3D) optical surface roughness analyzer (GT-X3 BASE, Bruker Co., Kalkar, Germany) at three points per specimen, and average roughness (Ra) values were calculated. Also the percentages of m-phase in the Katana and Rainbow groups were calculated by X-ray diffractometry (XRD; Ultima IV, Rigaku, Tokyo, Japan) of three specimens from each group that were not embedded in jigs. Scans were performed at 40 kV, 30 mA, from 20 to 80 ° with a step size of 0.02 ° for 0.6 s. XRD patterns were analyzed using the Rietveld refinement method, a whole pattern fitting procedure with MDI JADE (version 9.0, Material Date Inc., Livermore, CA, USA). The proportion of the m-phase was estimated from the relative integrated intensity of m(111), m($\bar{1}$11), and c,t(101) peaks. The mass fraction X_m of the m-phase percentage was calculated as follows (Garvie and Nicholson 1972).

$$X_m = \frac{I_m(\bar{1}11) + I_m(111)}{I_m(\bar{1}11) + I_m(111) + I_t(101)} \times 100$$

where $I_m(\bar{1}11)$ and $I_m(111)$ indicate the integrated intensities of the monoclinic peaks at 2θ = 28.2 ° and 2θ = 31.5 °, respectively. $I_t(101)$ denotes the integrated intensities of the tetragonal/cubic peaks at 2θ = $\tilde{3}0$ °. Quantification of the surface roughness and m-phase after wear analysis was performed in the Katana and Rainbow groups (n = 15, each).

3. Clinical study

An overview of the clinical study is shown in Figure 5.

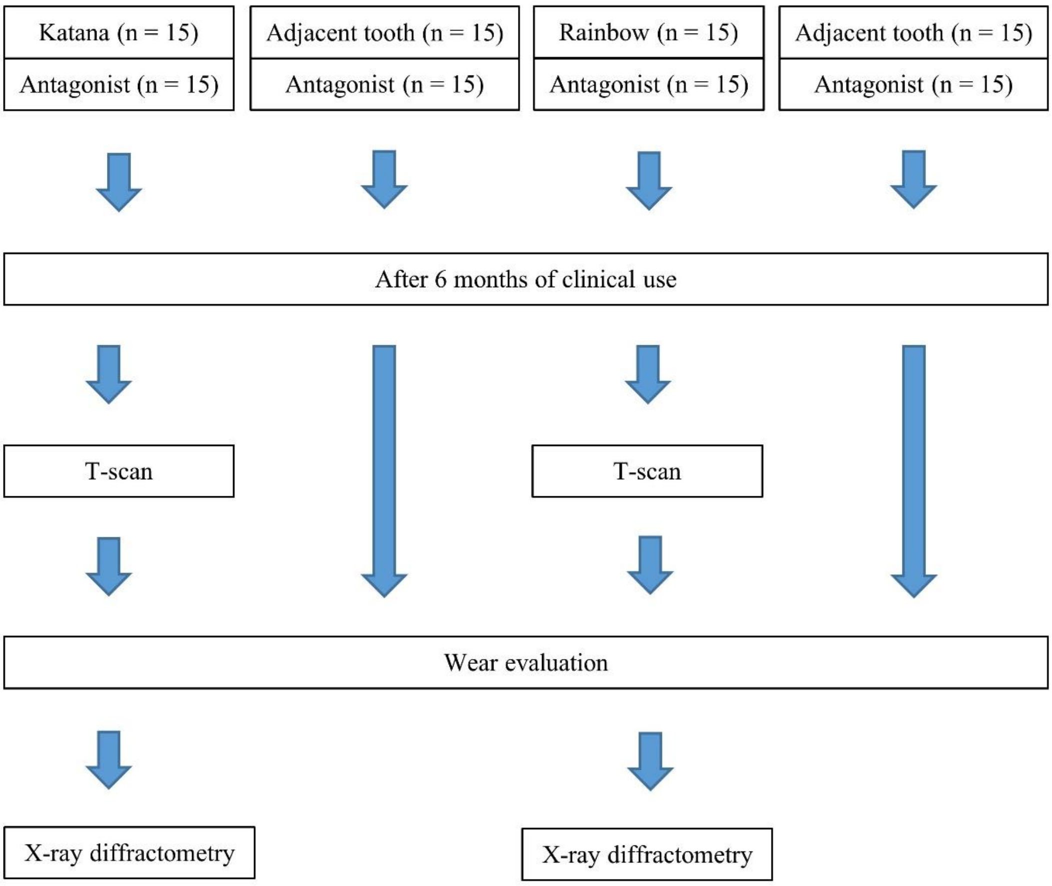

Figure 5. Schematic illustration of the clinical study.

3.1. Participants of the clinical study

Patients who visited the Department of Prosthodontics of the Yonsei University Dental Hospital between January and December 2016 were enrolled in the clinical study. The inclusion criteria were as follows: voluntary agreement for participation; requirement for single-crown restorations on first or second molar implants; age ≥ 19 years; absence of jaw-joint and other occlusal disorders and parafunctional habits such as bruxism; and presence of healthy natural opposing teeth. The exclusion criteria were as follows: inability to read the consent form; severe pathological findings in oral soft tissues; active lesions or symptoms on implants or restored teeth; unsuitability for the study, as adjudged by other clinicians. This study was approved by the institutional review board (approval number: 2-2015-0041), and written consent was obtained from all participants.

3.2. Crown fabrication and clinical procedures

After confirming stable osseointegration of implants, final impressions were taken for fabrication of ISFDPs, which comprised titanium custom abutments and zirconia crowns. Katana and Rainbow zirconia blocks (15 each) were randomly distributed, milled (Trione-Z, Dio Implant Co., Busan, Korea), and sintered in accordance with the manufacturer's instructions. Occlusal adjustment and polishing of prostheses on definitive casts were accomplished using zirconia polishing burs (Zirco master, Seichong Co., Seoul, Korea). The final prostheses did not undergo separate staining or glazing procedures. Additional adjustment and polishing of proximal and occlusal prosthesis surfaces with Zirco master were performed during delivery, if needed. The occlusions of the prostheses were set in harmony with surrounding teeth. Proper occlusal contacts were checked with an 8 μm aluminum foil (Shimstock foil, Union, Seoul, Korea). Occulsal interferences in centric and eccentric positions were removed. The prostheses were cemented with provisional cement (Temp-bond, Kerr Co., Orange, CA, USA) for re-evaluation.

3.3. Follow-up evaluation and occlusion analysis

Occlusal surfaces of the prostheses, antagonists, and adjacent teeth were recorded by closed-mouth impression with polyether (Monophase Polyether Impression Material, 3M ESPE Dental AG, Seefeld, Germany) at 1 week and 6 months after delivery. The impressions were scanned with the Identica Hybrid scanner to generate STL files.

Additionally, to determine the ratio of relative strength of prostheses in maximum intercuspation, the occlusion status of each patient was recorded using an occlusal diagnostic system (T-Scan 8, Tekscan Inc., South Boston, MA, USA) at 1 week and 6 months after prosthesis delivery. Differences in ratios between the two time points were calculated to identify changes in force transferred to prostheses.

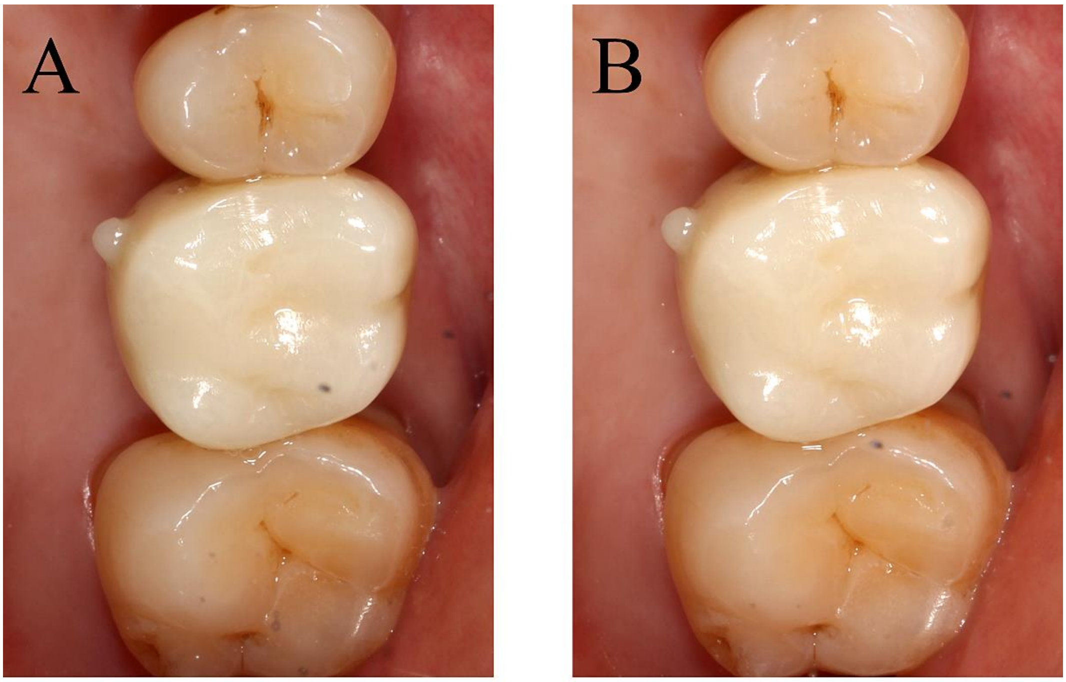

Figure 6. Intraoral photographs acquired at (A) 1 week and (B) 6 months from the day of prosthesis delivery. The prosthesis was stably maintained. No significant changes were observed visually.

3.4. Evaluation of occlusal wear

Using the Rapidform 2006 software, STL files of impressions acquired at 1 week and 6 months after prosthesis delivery were superimposed, and the occlusal surfaces of prostheses, antagonists, and premolars adjacent to prostheses and antagonists were extracted. Changes in mean and maximum vertical distances due to 6 months of wear were calculated for each of these components (Figure 7).

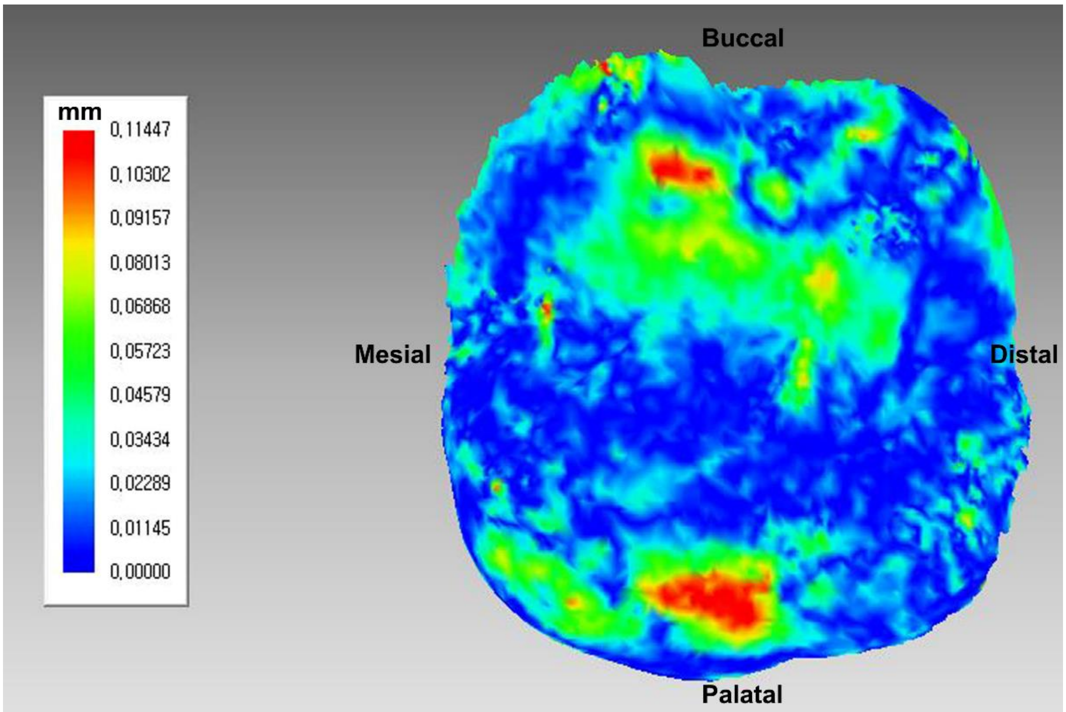

Figure 7. Representative image of clinical zirconia crown wear analysis. Red areas mean severe wear after 6 months of function.

3.5. Quantitative analysis of phase transformation

Closed-mouth impression and occlusal diagnosis were repeated at 6 months after prosthesis delivery. The existing prostheses were removed, and new prostheses were delivered. m-phase percentages of the removed prostheses were calculated by XRD analysis.

4. Statistical analysis

Statistical analysis was performed using IBM SPSS 23.0 (IBM Co., Armonk, NY, USA). Data normality was analyzed using the Kolmogorov–Smirnov test. Depending on the normality, Student's t-test or the Mann–Whitney test was used for data analysis. In vitro substrate wear was analyzed by one-way analysis of variance (ANOVA). Fisher's least significant difference (LSD) test was used for post-hoc analysis. In clinical studies, the effect of patient age, sex, and prosthesis type on wear was determined by multiple regression analysis. Significance was set at $\alpha = 0.05$.

III. Results

1. In vitro findings

The in vitro wear results are presented in Tables 2 and 3. The Katana, Rainbow and enamel groups exhibited significant differences in mean vertical wear of substrates ($p < 0.001$) and antagonists ($p = 0.017$; one-way ANOVA). There were significant differences in mean vertical wear of substrates between the Katana and Rainbow specimens ($p < 0.001$) and between Katana specimens and natural enamel ($p < 0.001$). The Katana specimens and natural enamel differed significantly in mean vertical wear of antagonists ($p = 0.004$; Fisher's LSD test).

The Katana and Rainbow specimens also differed significantly in Ra value ($p = 0.010$) after wear analysis and in mass fraction of m-phase percentages before ($p = 0.034$) and after ($p < 0.001$) wear analysis. However, neither group exhibited statistically significant differences in Ra values or m-phase percentages before and after wear test (Mann–Whitney test; Figures 8 - 9 and Tables 4 - 5).

Table 2. In vitro mean vertical wear evaluation results of substrate and antagonist. Data are presented as mean ± standard deviation.

Group		Mean vertical wear (μm)
Katana group	Katana	13.37 ± 3.29^{B}
	Antagonist	13.93 ± 2.14^{b}
Rainbow group	Rainbow	8.72 ± 2.01^{A}
	Antagonist	12.62 ± 2.94^{ab}
Enamel group	Enamel	9.32 ± 1.63^{A}
	Antagonist	11.26 ± 2.15^{a}

Superscript uppercase letters indicate differences in wear within a column among the three substrates. Superscript lowercase letters indicate differences in wear within a column among the three antagonists.

Table 3. In vitro maximum vertical wear evaluation results of substrate and antagonist. Data are presented as mean ± standard deviation.

Group		Maximum vertical wear (µm)
Katana group	Katana	100.48 ± 33.78 [A]
	Antagonist	145.96 ± 43.03 [a]
Rainbow group	Rainbow	92.70 ± 52.10 [A]
	Antagonist	156.00 ± 64.25 [a]
Enamel group	Enamel	82.72 ± 22.84 [A]
	Antagonist	156.32 ± 55.46 [a]

Superscript uppercase letters indicate differences in wear within a column among the three substrates. Superscript lowercase letters indicate differences in wear within a column among the three antagonists.

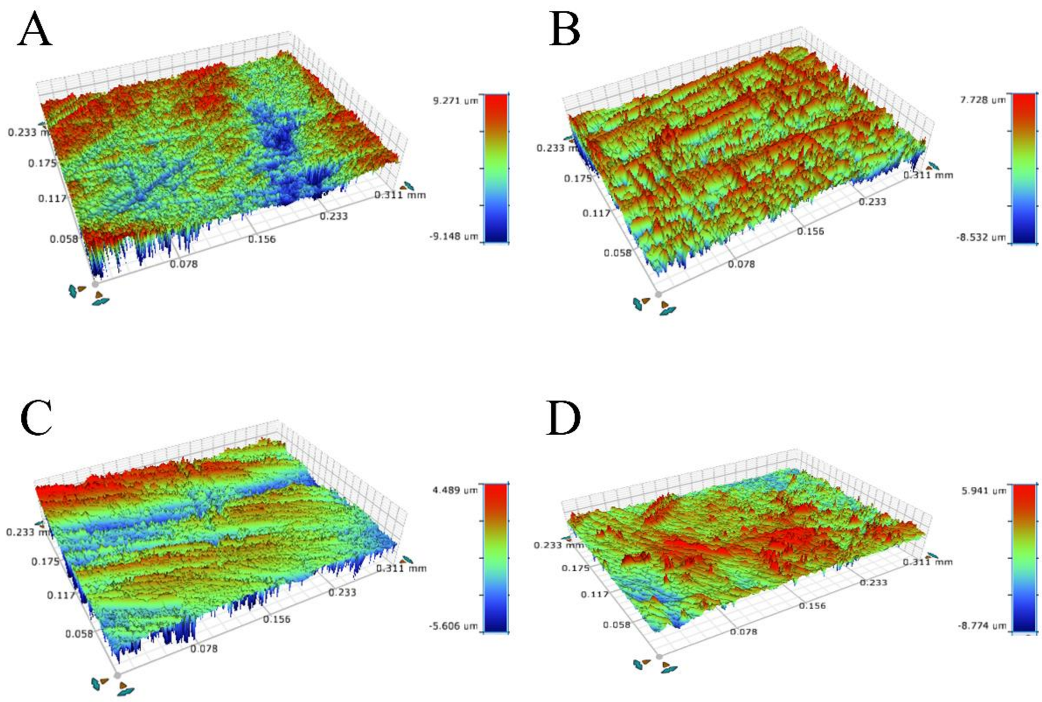

Figure 8. Representative surface roughness image of in vitro experiments. (A) Katana (Pretest), (B) Katana (After test), (C) Rainbow (Pretest), (D) Rainbow (After test), Katana group exhibited significantly greater surface roughness than Rainbow group. But neither group exhibited statistically significant differences in surface roughness before and after wear test.

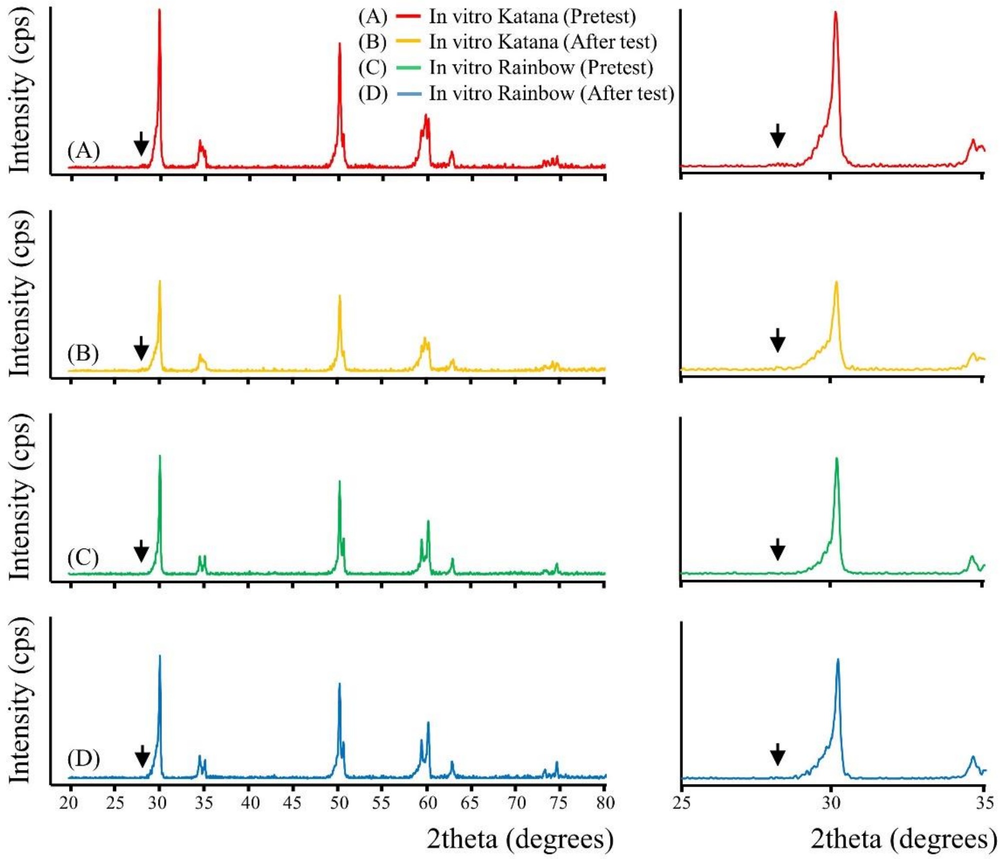

Figure 9. X-ray diffraction patterns in the Katana and Rainbow groups in the in vitro studies. Black arrows represent the differences of m-phase. Katana group exhibited significantly greater m-phase level than Rainbow group after wear test. But neither group exhibited statistically significant differences in m-phase level before and after wear test.

Table 4. In vitro evaluation results of surface roughness. Data are presented as median (interquartile range).

Group		Ra (μm)
Katana group	Pretest	1.06 (0.94 - 1.16) ^{Aa}
	After test	1.14 (0.90 - 1.32) ^{Ab}
Rainbow group	Pretest	0.78 (0.55 - 1.13) ^{Aa}
	After test	0.76 (0.57 - 1.08) ^{Aa}

Superscript uppercase letters indicate differences within the same group of zirconia within a column. Superscript lowercase letters indicate differences between two zirconia groups within a column. Ra; surface roughness.

Table 5. In vitro evaluation results of phase transformation. Data are presented as median (interquartile range).

Group		m-phase (%)
Katana group	Pretest	0.20 (0.15 - 0.20) ^{Ab}
	After test	1.10 (0.05 - 2.50) ^{Ab}
Rainbow group	Pretest	0.00 (0.00 - 0.00) ^{Aa}
	After test	0.00 (0.00 - 0.00) ^{Aa}

Superscript uppercase letters indicate differences within the same group of zirconia within a column. Superscript lowercase letters indicate differences between two zirconia groups within a column. m-phase; mass fraction percentage of monoclinic phase determined by X-ray diffractometry.

2. Clinical findings

The clinical study included 9 men and 6 women (mean age, 50.8 ± 12.1 years) in the Katana group and 11 men and 4 women (mean age, 49.2 ± 10.7 years) in the Rainbow group. Patient age and sex had no effect on enamel wear. Regression models for evaluation of wear index were found to be statistically unsuitable.

The clinical wear results are presented in Tables 6 - 9. Zirconia and natural tooth antagonists exhibited significant differences in mean ($p < 0.001$) and maximum ($p < 0.001$) vertical wear (paired t–test; Tables 6 - 7). Katana and Rainbow specimens exhibited a significant difference in mean vertical wear of prostheses ($p = 0.031$; Student's t-test). The Katana prostheses and their adjacent teeth exhibited significant differences in maximum vertical wear ($p = 0.013$; paired t-test) but not in mean vertical wear of antagonists. The Rainbow prostheses and their adjacent teeth exhibited significant differences in mean ($p = 0.002$) and maximum ($p < 0.001$) vertical wear of antagonists (paired t-test; Tables 8 - 9).

The Katana and Rainbow groups exhibited no significant differences in 6-month change in ratio of relative strength of prostheses in maximum intercuspation (mean, 0.23 ± 2.05% vs. mean, 0.15 ± 0.61%; $p = 0.501$; Student’s t-test) or in m-phase levels (median, 3.00% vs. median, 2.80%; $p = 0.493$; Mann–Whitney test; Figure 10 and Table 10).

Table 6. Comparison of mean vertical wear between the zirconia and natural tooth groups in the clinical study. Data are presented as mean ± standard deviation.

Group		Mean vertical wear (μm)
Zirconia group	Zirconia	28.30 ± 10.19 ^{A}
	Antagonist	32.39 ± 9.30 ^{B}
Natural tooth group	Adjacent tooth	25.09 ± 7.4 ^{A}
	Antagonist	25.51 ± 7.27 ^{A}

Superscript uppercase letters indicate differences between the zirconia and natural tooth groups within a column.

Table 7. Comparison of maximum vertical wear between the zirconia and natural tooth groups in the clinical study. Data are presented as mean ± standard deviation.

Group		Maximum vertical wear (μm)
Zirconia group	Zirconia	189.01 ± 48.75 [A]
	Antagonist	244.05 ± 60.13 [B]
Natural tooth group	Adjacent tooth	178.13 ± 30.60 [A]
	Antagonist	182.35 ± 44.77 [A]

Superscript uppercase letters indicate differences between the zirconia and natural tooth groups within a column.

Table 8. Clinical mean vertical wear evaluation results of substrate and antagonist. Data are presented as mean ± standard deviation.

Group			Mean vertical wear (µm)
Katana group	Zirconia	Katana	32.30 ± 11.72 ^{Ba}
		Antagonist	34.37 ± 11.12 ^{Aa}
	Natural tooth	Adjacent tooth	27.53 ± 9.10 ^{a}
		Antagonist	28.26 ± 8.29 ^{a}
Rainbow group	Zirconia	Rainbow	24.29 ± 6.57 $^{A\alpha}$
		Antagonist	30.41 ± 6.86 $^{A\beta}$
	Natural tooth	Adjacent tooth	22.65 ± 4.34 $^{\alpha}$
		Antagonist	22.76 ± 4.95 $^{\alpha}$

Superscript uppercase letters indicate differences in wear among the two zirconias and the two antagonists within the Katana and Rainbow groups. Superscript lowercase letters indicate differences in wear between zirconia and natural tooth within the Katana group. Superscript Greek letters indicate differences in wear between zirconia and natural tooth within the Rainbow group.

Table 9. Clinical maximum vertical wear evaluation results of substrate and antagonist. Data are presented as mean ± standard deviation.

Group			Maximum vertical wear (μm)
Katana group	Zirconia	Katana	190.95 ± 39.76 ^{Aa}
		Antagonist	222.86 ± 50.63 ^{Ab}
	Natural tooth	Adjacent tooth	177.03 ± 27.41 ^{a}
		Antagonist	187.82 ± 40.25 ^{a}
Rainbow group	Zirconia	Rainbow	187.07 ± 57.74 $^{A\alpha}$
		Antagonist	265.25 ± 62.96 $^{A\beta}$
	Natural tooth	Adjacent tooth	179.23 ± 34.44 $^{\alpha}$
		Antagonist	176.87 ± 49.68 $^{\alpha}$

Superscript uppercase letters indicate differences in wear among the two zirconias and the two antagonists within the Katana and Rainbow groups. Superscript lowercase letters indicate differences in wear between zirconia and natural tooth within the Katana group. Superscript Greek letters indicate differences in wear between zirconia and natural tooth within the Rainbow group.

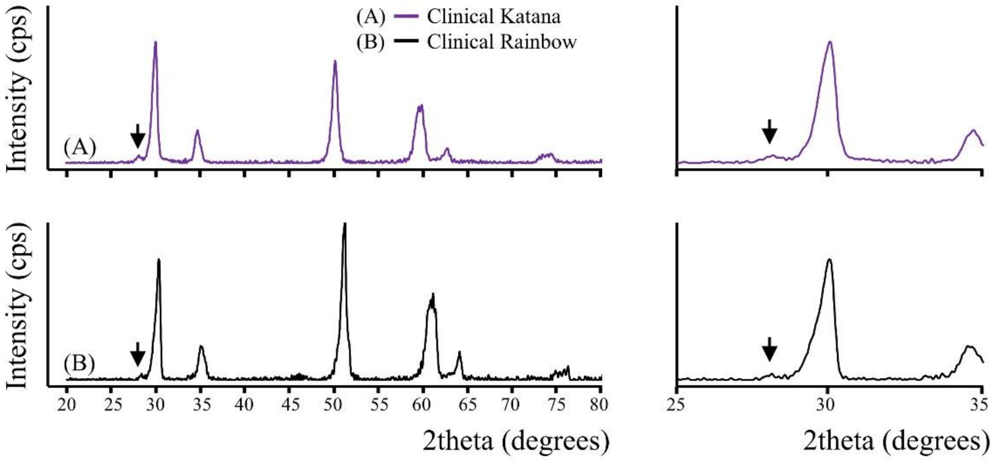

Figure 10. X-ray diffraction patterns in the Katana and Rainbow groups in the clinical studies. Black arrows represent the m-phase. Katana and Rainbow groups exhibited no significant differences in m-phase levels.

Table 10. Clinical evaluation results of phase transformation. Data are presented as median (interquartile range).

Group	m-phase (%)
Katana group	3.00 (1.80 - 4.35) ^{A}
Rainbow group	2.80 (0.70 - 4.30) ^{A}

Superscript uppercase letters indicate differences between two zirconia groups within a column. m-phase; mass fraction percentage of monoclinic phase determined by X-ray diffractometry.

3. Comparison of in vitro and clinical findings

Comparison of m-phase values of zirconia blocks in the clinical study and those after chewing simulation in the in vitro study revealed significant differences both in the Katana ($p = 0.022$) and Rainbow ($p < 0.001$; Mann–Whitney test) groups (Table 11).

Table 11. In vitro and clinical evaluation results of phase transformation. Data are presented as median (interquartile range).

Group		m-phase (%)
Katana group	In vitro (After test)	1.10 (0.05 - 2.50) ^{Ab}
	Clinical	3.00 (1.80 - 4.35) ^{Ba}
Rainbow group	In vitro (After test)	0.00 (0.00 - 0.00) ^{Aa}
	Clinical	2.80 (0.70 - 4.30) ^{Ba}

Superscript uppercase letters indicate differences within the same group of zirconia within a column. Superscript lowercase letters indicate differences between two zirconia groups within a column. m-phase; percentage of monoclinic phase determined by X-ray diffractometry.

IV. DISCUSSION

In the present study, we evaluated wear, surface roughness and phase transformation according to the use of polished Y-PSZ in both in vitro and clinical experiments. Within the limitations of this study, the null hypothesis that wear, surface roughness, and phase transformation would be similar for the two materials and between the in vitro and clinical experiments was rejected. Translucency increased zirconia, which reduced the need for an esthetic porcelain veneer, is used in dental field (Zhang 2014; Zhang et al. 2015). Research on the translucency increased zirconia different from the conventional 3Y-TZP is important.

Zirconia caused significantly greater wear of antagonists than did natural teeth (Tables 2, 6 and 7 - 9), which corresponds to the findings of previous clinical studies (Stober et al. 2014; Mundhe et al. 2015). However, in some previous in vitro studies, zirconia caused lesser wear of antagonists than did natural teeth (Janyavula et al. 2013; Burgess et al. 2014; Sripetchdanond and Leevailoj 2014). This discrepancy might be attributable to differences in surface roughness of zirconia specimens. Different polishing systems provide surfaces of different roughness on zirconia blocks (Bartolo et al. 2017; Park et al. 2017; Sim et al. 2017). However, the surface roughness of zirconia obtained using the Zirco master in the present study was somewhat greater than the surface roughness values reported in previous studies. This result is based on the fact that materials with rough surfaces cause more abrasive wear (Oh et al. 2002; Heintze et al. 2008; Passos et al. 2014). However, the present in vitro and clinical experiments provided similar tendencies of results over a 6-month period.

Katana specimens after wear test exhibited significantly higher Ra values than Rainbow specimens after wear test did (Table 4). Also, although there were no statistical significances in the in vitro and clinical studies, Katana substrates and prostheses induced greater mean vertical wear of antagonists than Rainbow substrates and prostheses did (Tables 2 and 8). The m-phase percentages were higher in the Katana specimens than in the Rainbow specimens (Table 5). The higher the phase transformation of zirconia is, the greater the surface roughness and the greater the wear of antagonist. Aging of zirconia leads to surface degradation, causing coarse surface that results in greater antagonistic wear (Chevalier et al. 1999; Oh et al. 2002; Heintze et al. 2008; Kohorst et al. 2012; Passos et al. 2014).

Katana specimens exhibited significantly greater mean vertical wear than Rainbow specimens did (Tables 2 and 8). Because of their higher Ra values and greater phase transformation, Katana specimens were expected to induce greater wear of antagonists than Rainbow specimens; however, there was no difference in wear of antagonists between the two groups. This suggests that not only surface roughness but other factors, too, can affect the wear of antagonists and zirconia. Since wear is influenced by various factors, it is challenging to explain the process on the basis of only a few factors (Ekfeldt and Oilo 1990; Sulong and Aziz 1990; Mair et al. 1996; Yip et al. 2004). However, although previous studies have shown that strength does not significantly affect wear, low-strength materials might cause greater wear because of increased surface roughness due to surface fracture (Kim et al. 2012).

Zirconia exhibited a significantly higher m-phase percentage in clinical experiments than in vitro (Table 11). According to International Organization of Standardization standards (ISO 13356. 2008), the allowed percentage of m-phase transformation is 25%; transformation levels obtained in the present study were below this limit (Lughi and Sergo 2010). The clinical effect of the small percentage of m-phase transformation of zirconia (2–3%) caused by aging has yet to be clarified. The present results indicate that both Katana and Rainbow zirconia blocks are clinically suitable in terms of aging over a period of 6 months.

There are two main reasons for the greater phase transformation of zirconia in clinical settings than in vitro. First, unlike in vitro experiments, the clinical experiments required additional occlusal adjustments during prostheses delivery, which might have caused phase transformation. Second, in vitro experiments were conducted in a contained environment, which can only limitedly reflect the actual oral conditions of patients; this difference was manifested in the greater overall wear in clinical experiments relative to that observed in vitro. These differences arise because of the following factors: the shape of in vitro substrate specimens is different from that of an actual crown; it is challenging to reproduce actual mandibular movement in a chewing simulator; and in vitro experimental wear corresponds to two-body wear, whereas actual intraoral wear is associated with three- and two-body wear (Harrison 1978). Although previous studies employed cyclic loading corresponding to 6 months of wear, the present wear and phase transformation results indicate that doubling the cyclic loading might help reproduce clinical results corresponding to 6 months of wear.

The Katana groups exhibited significantly higher m-phase levels than did the Rainbow groups, although the difference was statistical significant only in vitro (Tables 5 and 10). These differences in m-phase levels between the two groups throughout the study suggest that there might be a difference in susceptibility to aging between the two zirconias. The method of forming translucency increased zirconia involves decreasing Al_2O_3 content or increasing Y_2O_3 content in existing 3Y-TZP. However, decrease in Al_2O_3 content causes a decrease in aging resistance, while increase in Y_2O_3 content causes an increase in the cubic phase and decrease in the physical properties of zirconia, leading to a decrease in transformation toughening (Li and Watanabe 1997; Ross et al. 2001; Zhang 2014; Zhang et al. 2015). It is also known that surface roughness of zirconia is increased by aging (Passos et al. 2014). Relative to the Rainbow Shade Block, the Katana ML Blocks used in the present study possessed lower Al_2O_3 and higher Y_2O_3 content, which explains the greater m-phase levels and surface roughness of the latter.

There are three limitations to this study. First, since some subjects in clinical study had restorations on molars adjacent to zirconia prostheses, prostheses-adjacent premolars were used for wear analysis. Second, since zirconia crowns were delivered in the oral cavity, surface roughness could not be measured because of presence of other anatomic structures; for the same reason, the clinical XRD findings contained greater noise than did in vitro findings acquired with a flat specimen. However, the present results are meaningful in that the degree of LTD was confirmed by XRD analysis of zirconia that was actually used in the oral cavity. Third, this study involved both in vitro and clinical experiments on wear and phase transformation of Y-PSZ; however, the findings were acquired only over a 6-month period. Therefore, further long-term studies in this regard are required.

V. CONCLUSION

Based on the findings of this in vitro and clinical studies, the following conclusions were drawn:

1. The present findings suggest that zirconia caused significantly greater wear of opposing enamel than natural teeth did.
2. Phase transformation of zirconia occurred by clinical application.
3. The wear and degrees of phase transformation varied depending on the difference of zirconia.

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ABSTRACT (Korean)

이트리아 부분 안정화 지르코니아의 노화와 연관된 법랑질 마모에 대한 실험 및 임상 연구

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투명도가 향상된 이트리아 부분 안정화 지르코니아는 치과 수복 재료로 널리 사용되고 있다. 그러나, 이트리아 부분 안정화 지르코니아의 임상 적용에 따른 상변화 및 대합되는 법랑질의 마모량에 대해서는 아직까지 명확하게 밝혀지지 않고 있다. 본 연구의 목적은 연마된 이트리아 부분 안정화 지르코니아와 대합 법랑질의 마모를 평가하는 것이다. 또한, 이트리아 부분 안정화 지르코니아의 실제 사용에 따른 표면거칠기와 노화에 대해 실험적,

임상적으로 알아보고자 하는 것이다. 귀무가설은 실험 및 임상 연구에서 두가지 재료에 대해 마모, 표면거칠기 그리고 상변화가 비슷하다는 것이다.

실험 연구는 Katana 지르코니아 블록 (Katana ML Block, Kuraray Noritake Dental Inc. Aichi, Japan), Rainbow 지르코니아 블록과 (Rainbow Shade Block, Genoss Co., Suwon, Korea) 자연치의 법랑질을 (대조군) 사용하였고, 상악 소구치를 대합치로 사용하여 100,000 회 반복 하중을 시행하였다. 반복 하중 전과 후에 모든 시편을 스캔하여 마모량을 측정하였고, 지르코니아 시편에 대해서는 반복 하중 전과 후에 표면거칠기와 X 선 회절 분석을 통한 단사정계를 측정하였다.

임상 연구는 제 1 대구치 또는 제 2 대구치 임플란트에 단일 전장관 수복을 요하는 환자를 대상으로 시행하였다. 환자들의 임플란트는 Katana 또는 Rainbow 지르코니아를 이용하여 제작한 지르코니아 전장관으로 (각각 15 개) 수복하였다. 보철물 장착 1 주 및 6 개월 후 보철물, 대합치 및 인접치를 인상채득하여 교합면의 형태를 인기하여 마모량을 측정하였다. 또한 교합 진단 시스템을 (T-Scan 8, Tekscan Inc., South Boston, MA, USA) 이용해 최대 교두 감합위에서 보철물이 차지하는 상대적 힘의 비율을 측정하였다. 그리고, 구강 내에서 6 개월 사용 후 보철물을 제거하여 X 선 회절 분석을 통해 단사정계를 측정하였다.

6 개월간의 실험 연구와 임상 연구에서 지르코니아는 자연치에 비해 유의하게 대합치 법랑질의 더 많은 마모를 유발하였다. Katana 시편은 Rainbow 시편보다 유의하게 더 많은 마모가 발생했으며, 표면이 더 거칠게 나타났다. 그리고, 실험 연구보다 임상 연구에서 유의하게 대합치의 마모 및 지르코니아의 상전이가 많이 일어났다. Katana 그룹은 Rainbow 그룹에 비해 유의하게 더 많은 단사정계를 보였다.

결론적으로 임상 적용 시 지르코니아의 상전이가 발생하였으며, 지르코니아의 종류에 따라 마모량과 상전이 정도가 다르게 나타났다.

핵심 되는 말: 노화; 임상 연구; 상변화; 표면 거칠기; 마모; 지르코니아