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RESEARCH AND EDUCATION

Enamel wear and aging of translucent zirconias: In vitro and clinical studies

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ABSTRACT

Statement of problem. Zirconia is a widely used restorative material. However, phase transformation on clinical application of zirconia has not yet been studied.

Purpose. The purpose of this study was to evaluate the wear, surface roughness, and aging associated with polished translucent zirconia in both in vitro and clinical experiments.

Material and methods. In vitro experiments were performed with Rainbow and Katana zirconia blocks and natural tooth enamel as the control. They were subjected to 100 000 loading cycles with a maxillary premolar antagonist. All specimens were analyzed for wear, and the zirconia specimens were evaluated for surface roughness and monoclinic phase (m-phase) transformation by X-ray diffractometry before and after cyclic loading. The clinical study included participants who required single-crown implant-supported restorations replacing the first or second molar. The participants received Rainbow or Katana zirconia prostheses (n=15, each). For wear analysis, impressions of each prosthesis, antagonist, and adjacent tooth were made at 1 week and 6 months after crown delivery. The occlusal relationship of the crowns in maximum intercuspation was evaluated by using the T-Scan 8 occlusal diagnostic system. The degree of transformation of zirconia to the m-phase was measured by using X-ray diffractometry of the crowns after 6 months of use.

Results. Zirconia induced significantly greater enamel wear than the natural tooth control. Katana specimens exhibited significantly greater wear and surface roughness than the Rainbow specimens. The degrees of antagonistic wear and zirconia phase transformation in the clinical experiment were significantly greater than those in the in vitro experiment. The Katana groups showed significantly higher m-phase levels than the Rainbow groups.

Conclusions. Phase transformation of zirconia occurs within 6 months of clinical use, and the wear and degrees of phase transformation varied according to the zirconia product used. (J Prosthet Dent 2019;121:417-25)

Since its introduction in dentistry, monolithic zirconia has been widely used for implant-supported fixed dental prostheses and the restoration of natural teeth.[1-3] Early zirconia products had poor esthetics because of opacity, which required porcelain veneering on the zirconia core.[4] However, veneered porcelain is susceptible to chipping and delamination.[3] These problems have been overcome by the use of more esthetic monolithic zirconia. Nonetheless, the wear of antagonistic natural tooth enamel and the low-temperature degradation (LTD) of monolithic zirconia are controversial.

Wear is a complex phenomenon involving physical, chemical, and biological factors.[5-8] It is, therefore, challenging to reproduce complex oral conditions in vitro.[9,10] Excessive wear of teeth and restorative materials can lead to occlusal instability, decrease in vertical dimension and masticatory function, esthetic problems, and a poorly functioning oral maxillofacial system.[8,11] Therefore, the choice of restorative materials that replicate natural tooth wear, as tested with clinical studies, is important.[8]

Depending on the type of prosthesis, the wear of opposing enamel varies. Authors have reported that,

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Clinical Implications

Under actual use, translucent zirconias may behave differently compared with the suggestions of in vitro studies. Translucent zirconias cause greater wear of opposing enamel than do natural teeth. Also, clinical use of translucent zirconias causes phase transformation.

relative to porcelain, zirconia induces less wear on the antagonistic enamel of natural teeth.[2,11-17] Polished zirconia has been shown to induce less enamel wear than veneered or glazed zirconia.[17-19] However, most of these wear analysis studies have been in vitro experiments.

The LTD of zirconia further increases the complexity of the wear process.[20] Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP), which is widely used in zirconia restorations, maintains a tetragonal phase at room temperature.[21,22] However, on application of an 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% to 5% volume expansion that stops the progression of cracks because of compressive stress in a phenomenon termed transformation toughening.[23] However, in LTD, Y-TZP undergoes spontaneous tetragonal–m-phase transformation on the surface because of the presence of moisture and pressure in the oral cavity, the so-called aging of zirconia, leading to surface roughness and decreased strength.[24,25] Whether LTD causes strength degradation is controversial, and some studies have reported the opposite.[26,27] Opinions on the effect of strength of zirconia on the wear of opposing enamel also vary. Previously, stronger materials were deemed to cause greater wear of natural enamel.[28,29] However, recent studies have shown that the surface roughness of restorative materials, rather than their strength, has the dominant effect on the wear of opposing enamel.[18,20,28]

In Y-TZP, aging can affect the wear of opposing natural enamel because it results in decreased strength and increased surface roughness as a result of surface degradation.[20] However, few clinical studies have evaluated the effect of zirconia on enamel wear, and the authors are unaware of a study that has analyzed aging after the clinical application of zirconia.[2,11] Research on phase transformation after the application of zirconia in the oral cavity is necessary.[30] Moreover, experimental and clinical data are lacking on translucent zirconia,[31] which is used to provide increased translucency by controlling the content of alumina or yttria in the Y-TZP.[32,33] Further research is also required on the changes in the nature of translucent zirconia, including its physical and phase transformation properties.

The purpose of this study was to evaluate wear, surface roughness, and phase transformation with the use of polished translucent zirconia in both in vitro and clinical experiments. The null hypothesis was that wear, surface roughness, and phase transformation would be similar for the 2 materials and between the in vitro and clinical experiments.

Table 1. Materials investigated

Translucent zirconia	Manufacturer	Lot No.	Composition (wt.%)
Rainbow Shade Block	Genoss	15J05-04	ZrO_2, Y_2O_3 4–6%, $HfO_2 \leq 5\%$, $Al_2O_3 \leq 1\%$
Katana ML Block	Kuraray Noritake	DIBOT	ZrO_2, Y_2O_3 7.12–7.16%

MATERIAL AND METHODS

For the in vitro experiments, 3 types of substrate, Rainbow zirconia (Rainbow Shade Block; Genoss Co) (n=18), Katana zirconia (Katana Zirconia ML; Kuraray Noritake Dental Inc) (n=18), and natural enamel (n=15), were tested (Table 1).[34] Zirconia specimens were produced in a rectangular parallelepiped of 7×7×13-mm size (width×length×height). After sintering, the specimens were polished with zirconia polishing rotary instruments (Zirco Master; Seichong Co) in accordance with the manufacturer's instructions. Then, 15 each of Rainbow and Katana specimens were embedded in a custom device with acrylic resin (Fig. 1A). 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.[19] The flat part of the crown enamel was embedded in a custom device by using acrylic resin to contact the antagonist.

Extracted human maxillary premolars were used as antagonists. Premolars with fractured, worn, or exceptionally sharp cusps were excluded. After ultrasonic cleaning, the premolars were polished with pumice powder, homogenized,[19] and embedded in a device by using acrylic resin to ensure functional cusp contact with the substrate (Fig. 1B).

Wear analysis was performed by using a mastication simulator (Chewing Simulator CS-4.8; SD Mechatronik GmbH). The substrates (15 specimens per group) were mounted on the upper holder and the antagonists on the lower holder. The mastication simulation was performed under conditions of 2-mm-vertical and 2-mm-horizontal movement, 1.5-hz frequency, and a load of 49 N.[14,19] One hundred thousand cycles, corresponding to 6 months of function, were applied.[35] Each chamber was subjected to thermocycling between 5°C and 55°C.[14]

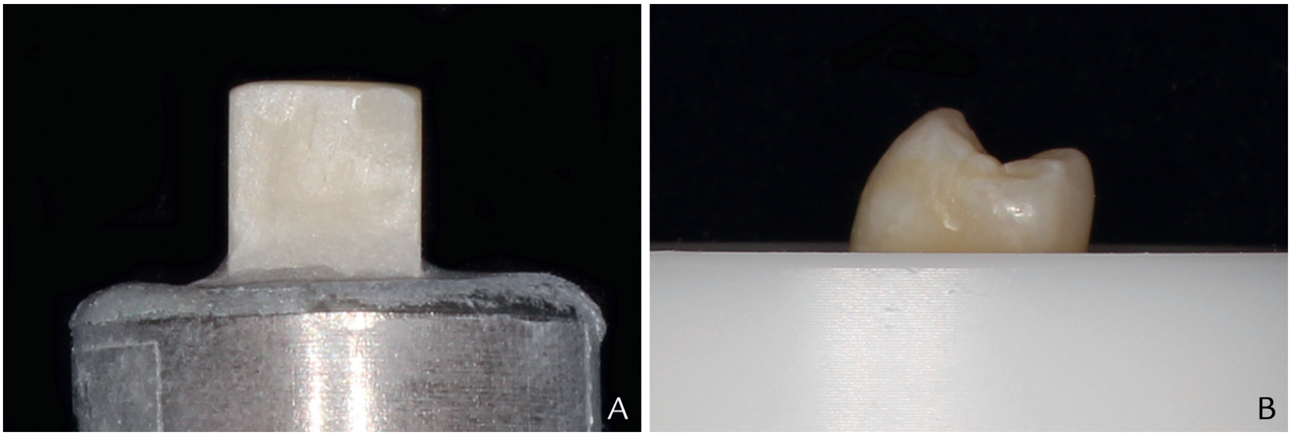

Figure 1. In vitro specimen for wear analysis. A, Zirconia substrate. B, Antagonist human maxillary premolar.

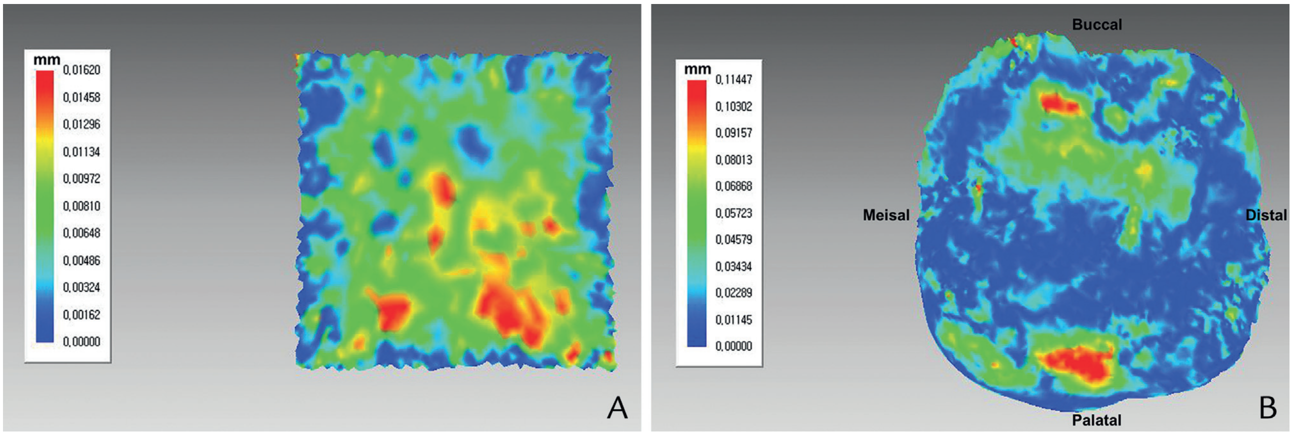

Figure 2. Representative image of in vitro and clinical wear analysis. A, In vitro zirconia substrate. B, Clinical zirconia crown; *red areas* indicate severe wear after 6 months of function.

The substrates and antagonists were scanned by using a blue light-emitting diode and a 50 ANSI-lumens desktop scanner (Identica Hybrid; Medit) with a resolution of mono 1.3 megapixel, 5.0 megapixel color camera, and an accuracy of 7 μm (ISO 12836) before and after wear analysis to generate standard tessellation language (STL) files. Using a 3-dimensional (3D) scanner software (Rapidform 2006; 3D Systems, Inc), STL files before and after wear analysis were superimposed, and the mean and maximum vertical differences caused by the wear test were calculated (Fig. 2A).

Before wear simulation, the surface roughness was measured by using a 3D optical surface roughness analyzer (Contour GT-X3 BASE; Brucker Co) at 3 locations per specimen, and average roughness (Ra) values were calculated. Also, the percentages of m-phase in the Rainbow and Katana groups were calculated by X-ray diffractometry (XRD) (Ultima IV; Rigaku) of 3 specimens from each group that were not embedded. Scans were performed at 40 kV, 30 mA, from 20 to 80 degrees with a step size of 0.02 degrees for 0.6 seconds. XRD patterns were analyzed by using the Rietveld refinement method, a whole pattern fitting procedure with software (MDI JADE v9.0; Materials Data, Inc). Quantification of the surface roughness and m-phase after wear analysis was

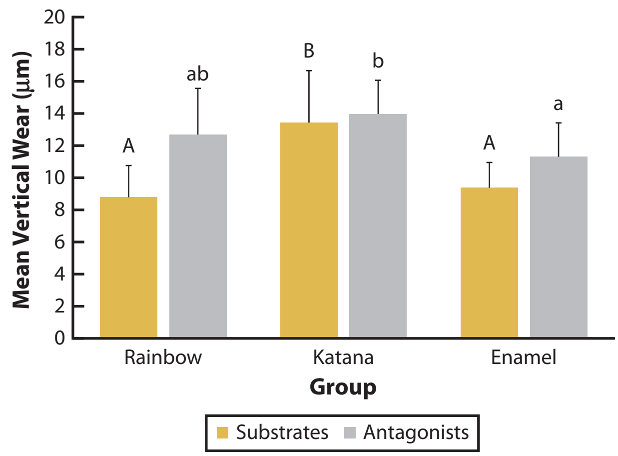

Figure 3. In vitro wear evaluation results of substrates and antagonists. Uppercase letters indicate differences in wear within row among 3 substrates. Lowercase letters indicate differences in wear within row among 3 antagonists.

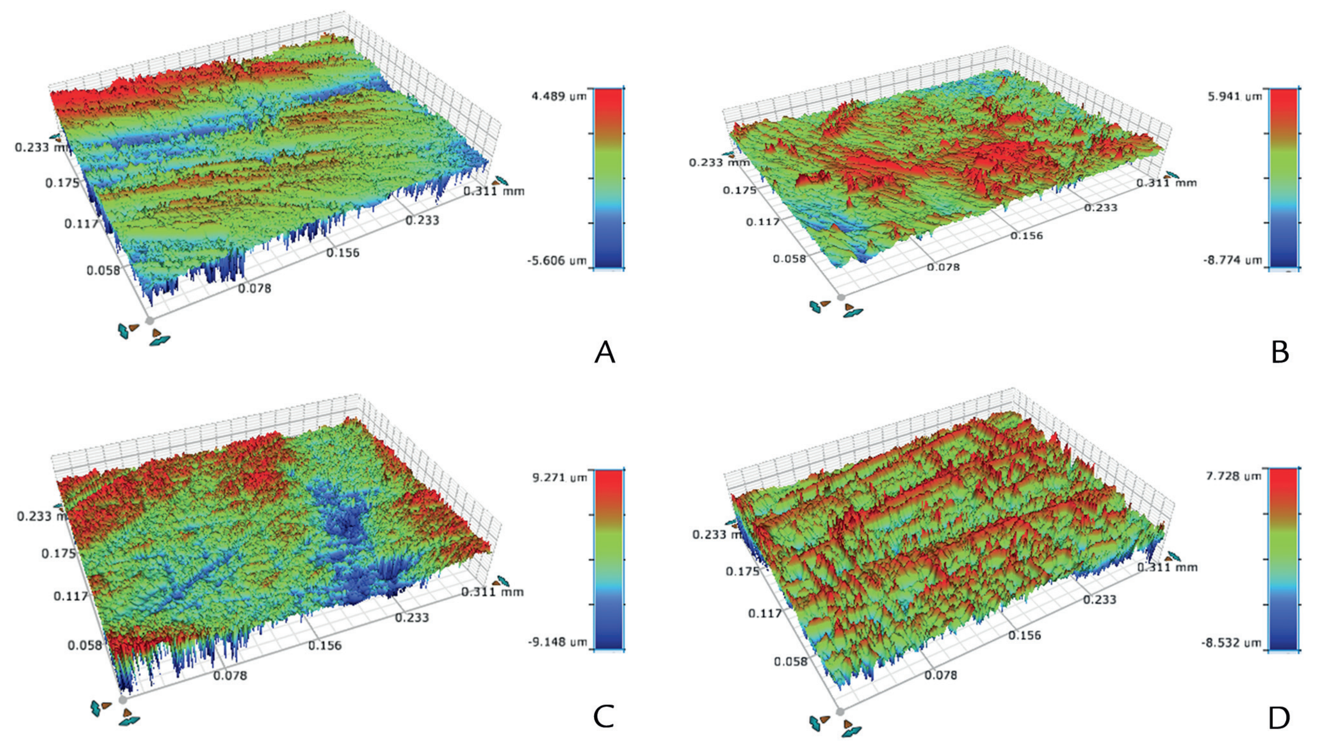

Figure 4. Representative surface roughness image of in vitro experiments. A, Rainbow (pretest). B, Rainbow (after test). C, Katana (pretest). D, Katana (after test); Katana group exhibited significantly greater surface roughness than Rainbow group. Neither group exhibited statistically significant differences in surface roughness before and after wear test.

performed with 15 specimens each in the Rainbow and Katana groups.

For the clinical wear experiments, patients who visited the Department of Prosthodontics of the Yonsei University Dental Hospital between January 2016 and December 2016 were recruited into the clinical study. This study was approved by the institutional review board (approval number: 2-2015-0041), and written consent was obtained from all participants. Inclusion criteria were voluntary agreement for participation; requirement for single-crown implant-supported restoration replacing the first or second molar; at least 18 years of age; absence of temporomandibular or other occlusal disorders and parafunctional habits such as bruxism; and the presence of healthy natural opposing teeth. The exclusion criteria were inability to read the consent form, severe pathological findings in oral soft tissues, active lesions or symptoms on implants or restored teeth, and unsuitability for the study as judged by other clinicians.

After confirming osseointegration of the implants, definitive impressions were made for the implant-supported prostheses, which comprised titanium custom abutments and zirconia crowns. Rainbow and Katana zirconia blocks (15 each) were randomly distributed by using computer-generated permuted block randomization with an allocation ratio of 1:1, milled (Trione-Z; Dio Implant Co.), and sintered in accordance with the manufacturer's instructions. Occlusal adjustment and polishing of the prostheses on definitive casts were accomplished by using zirconia polishing rotary instruments (Zirco Master; Seichong Co). The definitive prostheses were not stained or glazed. Additional adjustment and polishing of proximal and occlusal surfaces with zirconia polishing rotary instruments were performed during delivery if needed. The prostheses were cemented with interim cement (Temp-Bond; Premier Dental Products Co) to allow removal for re-evaluation.

A closed-mouth impression of the occlusal surfaces of the prostheses, antagonists, and adjacent teeth was made with polyether impression material (Monophase Polyether Impression Material; 3M ESPE) at 1 week and 6 months after delivery. The impressions were scanned (Identica Hybrid; Medit) to generate STL files.

In addition, to determine the occlusal contact relationship of the prosthesis in maximum intercuspation, an occlusal diagnostic system (T-Scan 8; Tekscan Inc) was used at 1 week and 6 months after prosthesis delivery. Differences between the 2 time points were calculated to identify changes in the force transferred to the prostheses.

STL files of impressions acquired at 1 week and 6 months after prosthesis delivery were superimposed with software (Rapidform 2006; 3D Systems, Inc), and the occlusal surfaces of prostheses, antagonists, and

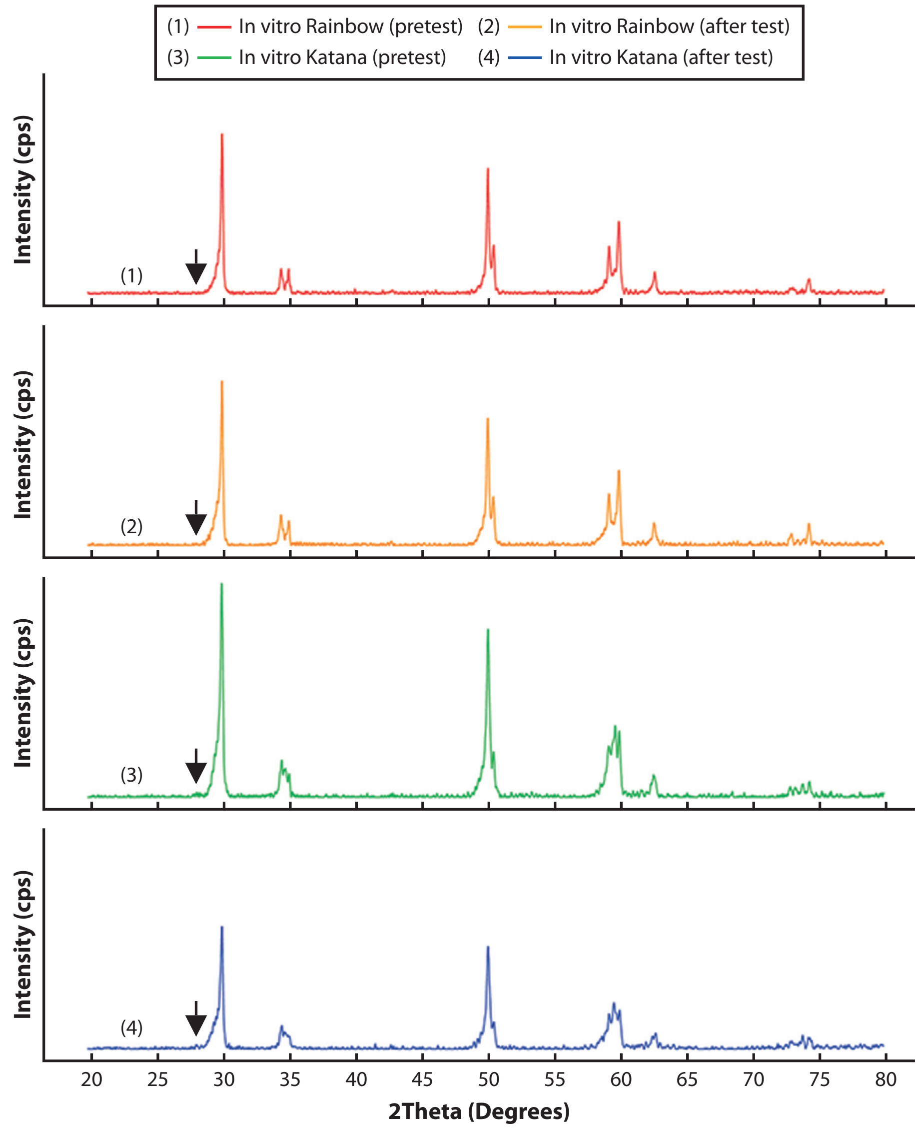

Figure 5. X-ray diffraction patterns in Rainbow and Katana groups from in vitro studies. *Black arrows* represent differences of m-phase. Katana group exhibited significantly greater m-phase level than Rainbow group after wear test. Neither group exhibited statistically significant differences in m-phase level before and after wear test.

premolars adjacent to prostheses and antagonists were extracted. Changes in mean and maximum vertical distances after 6 months of wear were calculated for each of these components (Fig. 2B).

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

Statistical analysis was performed by using software (IBM SPSS Statistics, v23.0; IBM Corp). The normality of the data was analyzed by using the Kolmogorov-Smirnov test. In the in vitro studies, substrate wear was analyzed by 1-way analysis of variance. The Fisher least significant difference test was used for post hoc analysis. The difference in Ra values according to the material and time points was analyzed by using the Student *t* test or paired *t* test. In the clinical study, the effect of patient age, sex, and prosthesis type on wear was determined by multiple regression analysis. Ra values and significant differences in 6-month change in ratio of relative strength of prostheses in maximum intercuspation was analyzed by using the Student *t* test, paired *t* test, or Mann-Whitney test (α=.05).

RESULTS

The in vitro wear results are presented in Figure 3. The Rainbow, Katana, and enamel groups exhibited significant differences in mean vertical wear of substrates (P<.001) and antagonists (P<.05). Significant differences were found in

Table 2. In vitro and clinical evaluation results of surface roughness and phase transformation

	Rainbow Group			Katana Group		
	In Vitro			In Vitro		
	Pretest	After Test	Clinical	Pretest	After Test	Clinical
Ra (μm)	0.78[Aa] (0.55-1.13)	0.76[Aa] (0.57-1.08)		1.06[Aa] (0.94-1.16)	1.14[Ab] (0.90-1.32)	
m-phase (%)	0.00 (0.00-0.00)	0.00 (0.00-0.00)	2.80 (0.70-4.30)	0.20 (0.15-0.20)	1.10 (0.05-2.50)	3.00 (1.80-4.35)

m-Phase, percentage of monoclinic phase determined by X-ray diffractometry; Ra, surface roughness. Values presented as median (interquartile range). Superscript uppercase letters indicate differences within same group of zirconia within rows. Superscript lowercase letters indicate differences between two zirconia groups within rows.

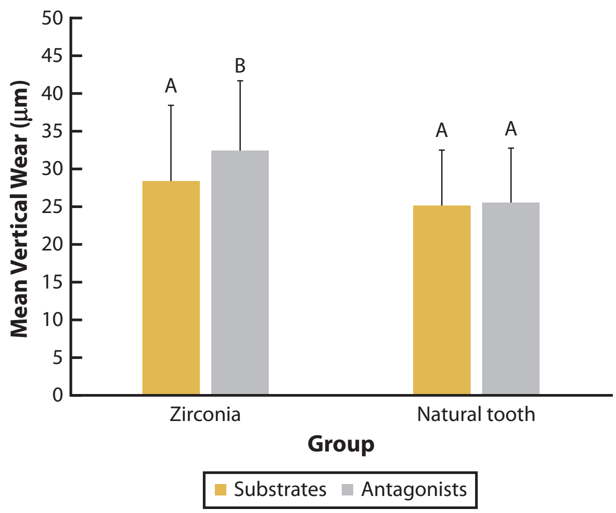

Figure 6. Comparison of wear between zirconia and natural tooth groups in clinical study. Uppercase letters indicate differences between zirconia and natural tooth groups within row.

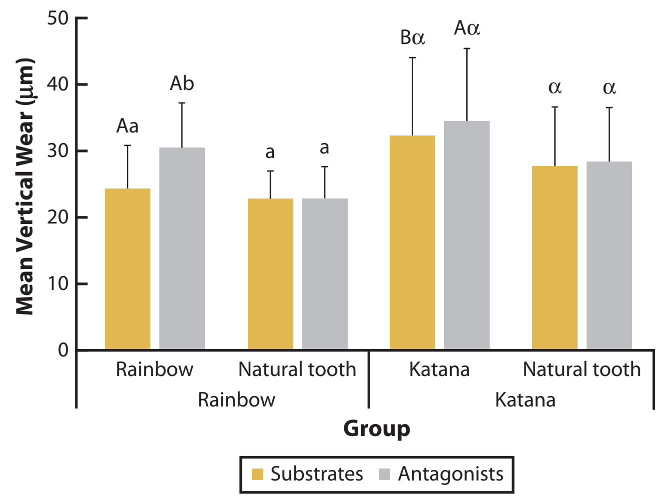

Figure 7. Clinical wear evaluation results of substrates and antagonists (mean ±standard deviation). Uppercase letters indicate differences in wear among 2 zirconias and 2 antagonists within Rainbow and Katana groups. Lowercase letters indicate differences in wear between zirconia and natural tooth within Rainbow group. Greek letters indicate differences in wear between zirconia and natural tooth within Katana group.

mean vertical wear of substrates between the Rainbow and Katana specimens (P<.001) and between Katana specimens and natural enamel (P<.001). The Katana specimens and natural enamel differed significantly in the mean vertical wear of antagonists (P<.01).

The Ra values between the materials of the Rainbow and the Katana specimens were significantly different after the wear analysis (P<.05). However, Ra values the same material showed no significant difference between before and after wear analysis (Figs. 4, 5; Table 2).

The clinical study included 11 men and 4 women (mean age, 49.2 ±10.7 years) in the Rainbow group and 9 men and 6 women (mean age, 50.8 ±12.1 years) in the Katana 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 Figures 6 and 7. Zirconia and natural tooth antagonists exhibited significant differences in mean (P<.001) vertical wear (Fig. 6). Rainbow and Katana specimens exhibited a significant difference in mean vertical wear of prostheses (P<.05). The Rainbow prostheses and their adjacent teeth exhibited significant differences in mean (P<.01) vertical wear of antagonists. The Katana prostheses and their adjacent teeth not exhibited significant differences in mean vertical wear of antagonists (Fig. 7).

The Rainbow and Katana groups exhibited no significant differences in 6-month change in the ratio of occlusal contact in maximum intercuspation (mean, 0.15 ±0.61% versus mean, 0.23 ±2.05%; P>.05) or in m-phase levels (median, 2.80% versus median, 3.00%; P>.05; Fig. 8, Table 2). A comparison of m-phase values of zirconia blocks in the clinical study and those after mastication simulation in the in vitro study revealed significant differences both in the Rainbow (P<.001) and Katana (P<.05) groups (Table 2).

DISCUSSION

Within the limitations of this study, the null hypothesis that wear, surface roughness, and phase transformation would be similar for the 2 materials and between the in vitro and clinical experiments was rejected. Wear, surface roughness, and phase transformation of polished translucent zirconia were evaluated in both in vitro and clinical experiments. Translucent zirconia has become popular as a dental restorative as it reduces the need for an esthetic porcelain veneer.[32,33] The behavior of translucent zirconia may be different from the conventional 3Y-TZP; therefore, research is important.

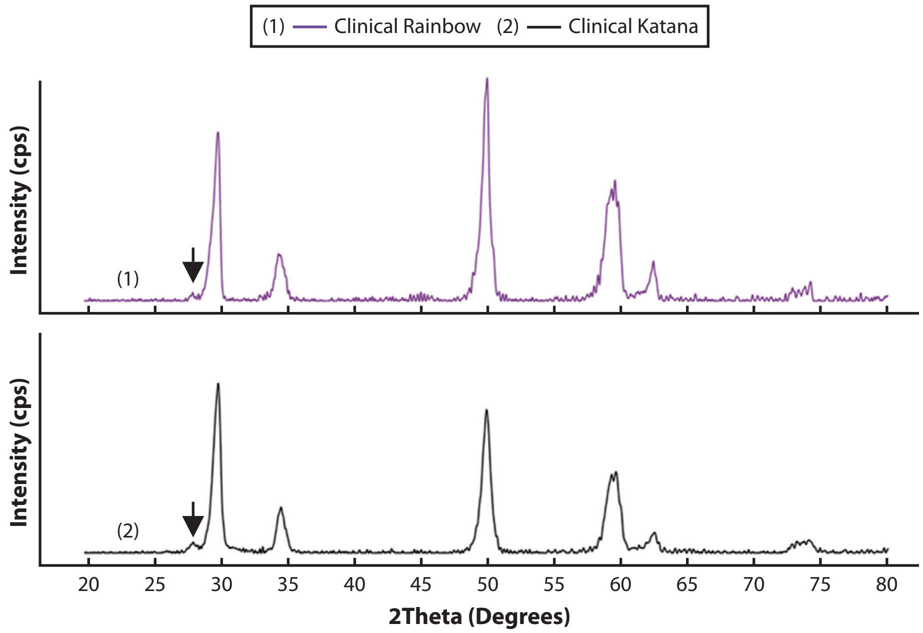

Figure 8. X-ray diffraction patterns in Rainbow and Katana groups in clinical studies. *Black arrows* represent difference of m-phase. Rainbow (median, 2.80%) and Katana (median, 3.00%) groups exhibited no significant differences in m-phase levels.

Zirconia caused significantly greater wear of antagonists than did natural teeth (Figs. 3, 6), which corresponds to the findings of previous clinical studies.[2,11] However, in some previous in vitro studies, zirconia caused less wear of antagonists than did natural teeth.[12,16,19] This discrepancy might be attributable to differences in surface roughness of zirconia specimens. Different polishing systems provide surfaces of different roughness on zirconia blocks.[36-38] However, the surface roughness of zirconia obtained by using the polishing system (Zirco master; Seichong Co) in the present study was somewhat greater than the surface roughness values reported in previous studies, indicating that materials with rough surfaces cause more abrasive wear.[18,20,28] However, the present in vitro and clinical experiments tended to provide similar results over a 6-month period.

Katana specimens exhibited significantly greater mean vertical wear than did Rainbow specimens (Figs. 3 and 7). Because of their higher Ra values and greater phase transformation, Katana specimens were expected to induce greater wear of antagonists than Rainbow specimens; however, no difference was found in wear of antagonists between the 2 groups. This suggests that not only surface roughness but also other factors can affect the wear of antagonists and zirconia. Because wear is influenced by various factors, explaining the process on the basis of only a few factors is challenging.[5-8] However, although previous studies have shown that strength does not significantly affect wear, lower strength materials might cause greater wear because of increased surface roughness due to surface fracture.[14]

Zirconia exhibited a significantly higher m-phase percentage in clinical experiments than in vitro (Table 2). According to the International Organization of Standardization standard ISO 13356:2008,[39] the allowed percentage of m-phase transformation is 25%; transformation levels obtained in the present study were below this limit.[40] The clinical effect of the small percentage of m-phase transformation of zirconia (2% to 3%) caused by aging has yet to be clarified. The present results indicate that both Rainbow and Katana zirconia blocks are clinically suitable in terms of aging over a period of 6 months.

There are 2 main reasons for the increased phase transformation of zirconia in clinical settings than in vitro. First, unlike in vitro experiments, the clinical experiments required occlusal adjustments during prostheses delivery, which might have caused phase transformation. Second, in vitro experiments were conducted in a contained environment, which can only reflect actual oral conditions to a limited degree. Differences between the in vitro testing and the clinical situation include the shape of in vitro substrate specimens which was different from that of an actual crown as it is challenging to reproduce actual mandibular movement in a mastication simulator. In addition, the in vitro experimental wear corresponded to 2-body wear, whereas actual intraoral wear is associated with both 3- and 2-body wear.[41] Although previous studies used 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 (Table 2). These differences suggest a possible difference in susceptibility to aging between the 2 zirconias. The method of forming translucent zirconia involves decreasing the Al_2O_3 content or increasing the Y_2O_3 content in existing 3Y-TZP. However, a decrease in Al_2O_3 content causes a decrease in aging resistance, while an increase in Y_2O_3 content causes an increase in the cubic phase and a decrease in the physical properties of zirconia, leading to a decrease in transformation toughening.[32,33,42,43] In addition, surface roughness of zirconia is increased by aging.[20] 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 Katana ML Blocks.

This study has limitations. Some participants in the clinical study had restorations on molars adjacent to zirconia prostheses, and prosthesis-adjacent premolars were used for wear analysis. Surface roughness could not be measured intraorally because of the presence of other anatomic structures; for the same reason, the clinical XRD findings contained greater noise than did in vitro findings acquired from a flat specimen. However, the present results are meaningful in that the degree of LTD was confirmed by XRD analysis of zirconia used in the oral cavity. In addition, this study involved both in vitro and clinical experiments on wear and phase transformation of translucent zirconia; however, the findings were acquired only over a 6-month period. Therefore, longer term studies are indicated.

CONCLUSIONS

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

1. Zirconia caused significantly greater wear of opposing enamel than natural teeth did.
2. Phase transformation of zirconia occurred within 6 months of clinical application.
3. The wear and degree of phase transformation varied depending on the zirconia material used.

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Noteworthy Abstracts of the Current Literature

Implant inclination and cantilever length are not associated with bone loss in fixed complete dentures: A prospective study

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Purpose. To investigate whether the inclination of the most distal implant and the cantilever length influence marginal bone loss in implant-supported fixed complete dentures (ISFCDs).

Material and methods. A novel method using computed tomography images was developed to measure the mesiodistal implant inclination. The cantilever length was measured during ISFCD fabrication. Radiographs were obtained after ISFCD installation at 1 and 3 years after loading.

Results. A total of 30 subjects with 62 implants were included. Accumulated marginal bone loss was 0.35 ± 0.49 mm. No significant association was found between marginal bone loss and cantilever length or implant inclination.

Conclusions. Implant inclination and cantilever length do not seem to affect marginal bone loss.

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