Dental Materials Journal 2013; 32(5): 793–800

Effects of coloring agents applied during sintering on bending strength and hardness of zirconia ceramics

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The effects of coloring agents (Vita in-ceram 2000 YZ coloring liquid (VL) and IPS e.max ZirCAD (IS)) and shades (1, 3, and 5) applied during sintering on the bending strength and fracture toughness of zirconia ceramics was examined. No differences in the bending strength or fracture toughness were observed for the type of coloring agent used. Moreover, the bending strength and Vickers hardness of the zirconia ceramics decreased, while the crack length and fracture toughness did not change with the different coloring agents. The marginal borders of the indentations formed were clear and linear, and no damage, including chipping, was observed. Therefore, clinical application of zirconia ceramics can be recommended because the coloring agents and shades applied during sintering have the same effect as an opaque layer and cause no significant deterioration of the mechanical properties of the zirconia ceramics.

Keywords: Zirconia Ceramics, Sintering, Bending strength, Fracture toughness

INTRODUCTION

Zirconia ceramics (henceforth abbreviated as "zirconia") are biomaterials that exhibit superior strength and toughness[1-4]. In the field of dentistry, zirconia is applied to frameworks for esthetic crown prostheses including all-ceramic dental restorations in a clinical setting. Zirconia-based frameworks have recently attracted increased attention as materials that exhibit functional performance and strength equal to that of metal frameworks because simple crowns and even bridges for one to two missing teeth can be restored using zirconia-based frameworks[5-11].

Esthetic restorations in dentistry are performed by cutting the tooth enamel and attaching crown and bridge. Therefore, the concept of esthetic restoration is different from that of minimal intervention (MI), which is used for regenerative soft tissue and organs. In recent developments in MI esthetic restoration, enamel is not cut as soon as possible, and the cut enamel and dentinal surfaces are bonded with adhesive. As a result, the increased acid resistance of the tooth substance can prevent secondary caries due to leakage and prevent discoloration of margins after restoration with ceramic and resin composites[12-14].

If layering porcelains[15,16] and hybrid resin[17-20] for dental crowns can be established as techniques that provide strong adhesion to zirconia-based frameworks, they can guide the selection of safe, reliable treatment measures based on using prosthetic crowns to apply zirconia in esthetic restoration. If restoration materials are used in compliance with demand from patients, metal-free crowns and bridges will have wider application. In addition, technological developments based on computer-aided design/computer-aided modeling (CAD/CAM) systems can lead to higher quality restoration devices, standardized processing accuracy, a simplified production process, and an improved laboratory environment for dental technicians[21,22]. Although it has been difficult to reproduce natural tooth color on frameworks because of high luminosity (white color) and flexibility, elaborate shades can be produced by staining the surface of the framework with coloring agents, achieving high esthetic quality even for the anterior teeth[23,24]. Although it has previously been reported that the mechanical properties of zirconia-based frameworks deteriorate after colors are reproduced on the frameworks by chemical conjugation formed during burning after the frameworks have been sintered in a solution of coloring agents[25-27], there are few reports on the mechanism of the deterioration of the mechanical properties. It is also necessary to determine the changes in the properties of the zirconia surface due to the use of coloring agents applied during sintering.

Few studies have investigated degradation and much is yet to be understood about this process; therefore, no there is no consensus about this process. We conducted basic physical experiments to collect evidence for understanding the mechanism underlying degradation.

With the aim of clarifying how coloring and colorants during sintering affect the flexure strength and fracture toughness of zirconia ceramics, the present study comprised a 3-point bending test of colored samples in which the load and bending strength were measured up to the point of fracture. Changes in surface texture of the colored samples were also observed, along with scanning electron microscopy (SEM) observation of the form of indentations and shape of cracks.

Color figures can be viewed in the online issue, which is available at J-STAGE.

Received Apr 12, 2013: Accepted Jul 11, 2013
doi:10.4012/dmj.2013-110 JOI JST.JSTAGE/dmj/2013-110

MATERIALS AND METHODS

Materials

The materials used for our experiments were samples of zirconia Y-TZP (Kavo Everest®, Zirconium Soft, Kavo, Biberach, Germany). Vita in-ceram 2000 YZ coloring liquid (Vita Zahnfabrik, Bad Sackingen, Germany (abbreviated as VL)), and IPS e.max ZirCAD coloring liquid (Ivoclar Vivadent, Schaan, Liechtenstein, (abbreviated as IS)) were used for coloring agents. The following coloring liquids were selected for obtaining the different shades: lightest shades, VL-1 and IS-1; middle shades, VL-3 and IS-3; and dark shades, VL-5 and IS-5. The composition of Y-TZP was 3 wt% Y_2O_3 and 97 wt% ZrO_2. The compositions of the materials used in this study are shown in Table 1.

Specimen preparation

Figure 1 shows the zirconia block (a), zirconia specimen (b) and dimension of the test piece (c). The test bars used for the bending test were in the form of 2.0×5.0×25.0 mm plates according to ISO standard (ISO 6872). Dimensional correction at a shrinkage rate of 20% was applied to a 20×20×40 mm partially sintered zirconia (Kavo Everest® Zirconium Soft, Biberach, Germany) block, and a 2.6×6.2×30.2 mm form was clipped using a low-speed cutting machine (ISOMET, Springfield, USA). In addition, the edge faces of the test bars were processed to be parallel (0.05 mm), and the bulging parts were cut off. The test bars were subsequently immersed in coloring liquids for 2 min, rinsed with purified water, and naturally air-dried after excess water was wiped off with a sheet of absorbent paper (Kimwipe wipers s-200, Cresia, Tokyo, Japan). The test bars were then sintered in a firing furnace (Everest Therm, Biberach, Germany) for 10 h until the heating temperature rose to 1450°C, according to the manufacturer's recommended conditions described in Fig. 2. Figure 3 shows the completed specimens.

Measurement methods

1. Three-point bending test

Three-point bending strength (BS) was measured using the three-point bending test method, as described in the ISO standard 6872: 2008 for dental ceramics. Load was applied at a crosshead speed of 0.5 mm/min and at a 15-mm support span. BS (in MPa) is given by

$$BS=3wl/2bd^2,$$

where w is the fracture load in N, l is the supporting width in mm, and b and d are the width and height of test specimen in mm, respectively.

2. Fracture toughness test

Fracture toughness (expressed in K_{IC}) was measured using the indentation fracture method. By means of a Vickers hardness testing machine (AVK-A, Akashi Co., Kanagawa, Japan), a diamond indenter was pressed into the specimen surface at a load of 196 N (20 kgf) for 15 s. The size of the impression left in the specimen surface was then measured after the load was removed. Three different points were measured for each specimen, which thus required three impressions to be produced in each specimen. The measured value for each specimen was the average value obtained from the three measurements, and the fracture toughness was calculated using the following equation:

Table 1 Name, code, manufacture, Lot No., and zirconia composition

Name	Code	Manufacturer	Lot number	Composition
Zirconium Soft	ZS	Kavo	100921645	3 wt%Y, 97 wt%ZrO_2
Vita In-Ceram 2000	VL1		26200	
YZ Coloring Liquid	VL3	VITA	24030	—
LL 1,3,5	VL5		16870	
IPS e.max ZirCAD	IS1	Ivoclar	L18684	
Shade 1,3,4	IS3	Vivadent	L08537	—
	IS5		L05159	

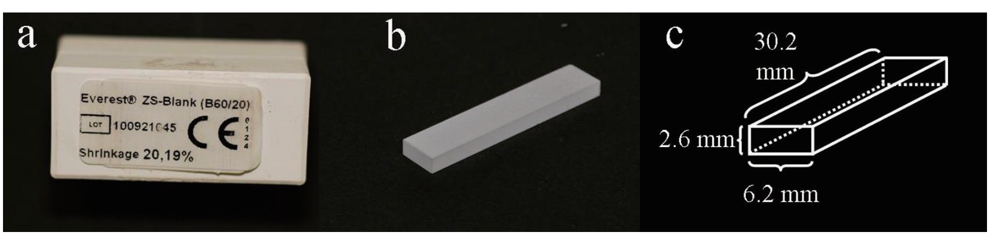

Fig. 1 Zirconia block (a) and zirconia test piece (b) and dimension of the test piece (c) used in this study.

$K_{I}c = 0.203(c/a)^{-3/2}Ha^{1/2}$,

where $K_{I}c$ is the fracture toughness ($MPa \cdot m^{1/2}$), a is one-half the diagonal of the impression (μm), c is one-half the crack length (μm), and H is the Vickers hardness.

3. SEM examination

After the indentation fracture test, the fractured specimen surfaces were observed using a scanning electron microscope (SEM S-400, Hitachi Co., Ltd., Tokyo, Japan) at original magnification ×300 and at a 5-kV acceleration voltage.

Methods

A two-way analysis of variance (ANOVA) was performed for bending strength and fracture toughness. The test included a combination of 6 levels in which coloring agent types (VL and IS) were denoted as factor A and coloring agent shades (shades 1, 3, and 5) were denoted as factor B. The test was repeated 10 times, and a total of 60 tests were randomly conducted. After the obtained results were confirmed as homoscedastic, they were statistically processed in a two-way analysis ($p<0.01$). Tukey's multiple comparison tests were conducted on the results that were significantly different.

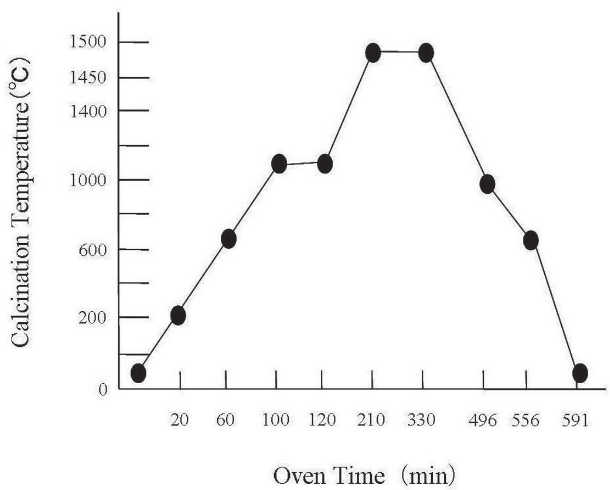

Fig. 2 Firing schedule.

Fig. 3 Test specimens used in this study.

RESULTS

Bending strength, Vickers hardness, crack length and fracture toughness of zirconia

As a result of the two-way analysis of variances calculated from the bending strength, hardness, crack length, and fracture toughness, main effect B (coloring agent shade) showed a significant difference in bending strength and hardness with a 99% confidence level, and main effect A (coloring agent type) and interaction A×B showed no significant difference. All factors showed no significant difference in crack length and fracture toughness.

Figure 4 shows the effect of the coloring agents on bending strength. Although there was a significant difference between shade1 and shades 3, and 5, there was no difference between shades 3 and 5. The mean bending strength was 921, 592, and 585 MPa for VL-1, 3, and 5, respectively, and 710, 631, and 652 MPa for IS-1, 3, and 5, respectively. Figure 5 shows the effect of the coloring agents on Vickers hardness. Although there was a significant difference between shades 1 and 3 and shade 5, there was no significant difference between shades 1 and 3. The mean Vickers hardness was 1340, 1330, and 1267 HV for VL-1, 3, and 5, respectively, and 1351, 1326, and 1281 HV for IS-1, 3, and 5, respectively. Figure 6 shows the effect of the coloring agents on crack length. The mean crack length was 197, 193, and 201 μm for VL-1, 3, and 5, respectively, and 193, 193, and 199 μm for IS-1, 3, and 5, respectively. Figure 7 shows the effect of the coloring agents on fracture toughness. The mean fracture toughness was 18.51, 18.60, and 18.49 $M \cdot Pam^{1/2}$ for VL-1, 3, and 5, respectively, and 19.04, 19.00, and 18.62 $M \cdot Pam^{1/2}$ for IS-1, 3, and 5, respectively.

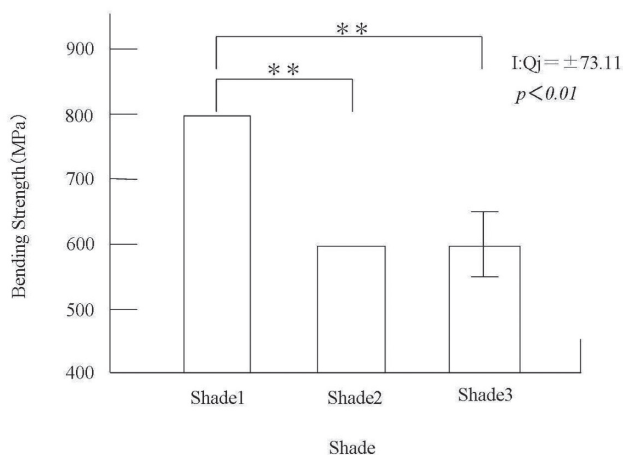

Fig. 4 Effect of coloring agents on bending strength.

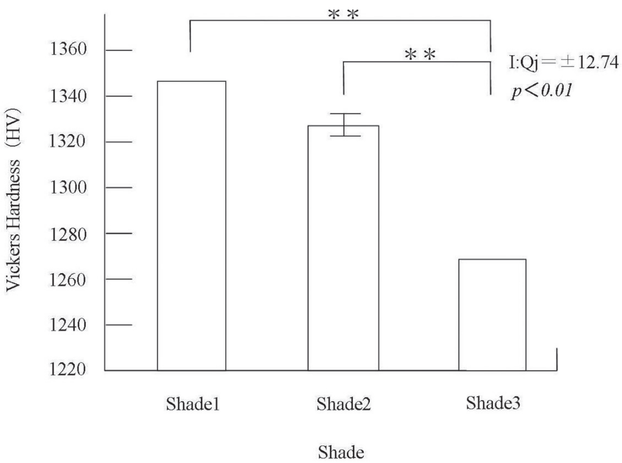

Fig. 5 Effect of coloring agents on Vickers hardness.

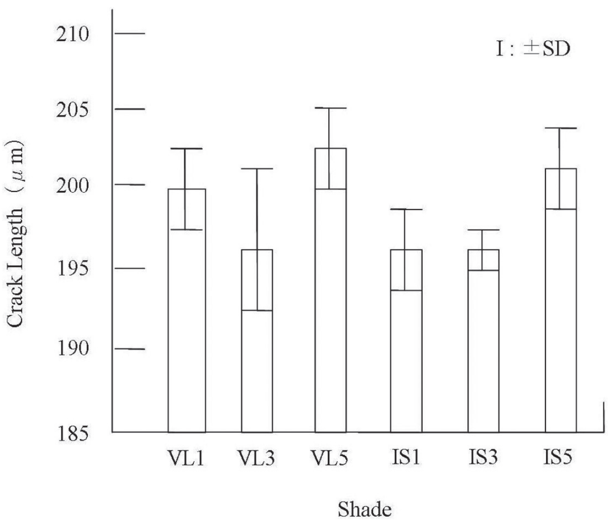

Fig. 6 Effect of coloring agents on crack length.

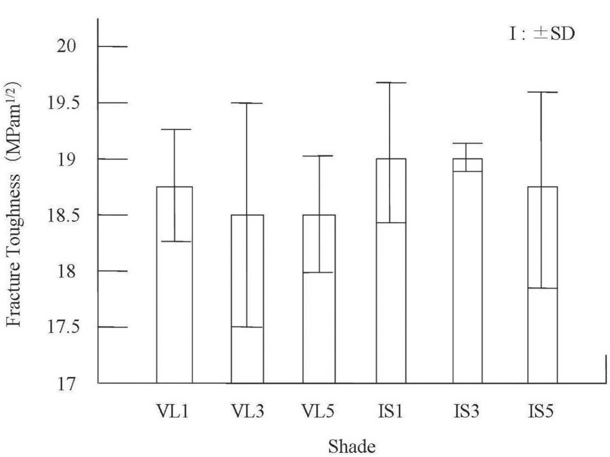

Fig. 7 Effect of coloring agents on fracture toughness.

SEM observation

Figure 8 shows the SEM images of the surface of the test bars that were immersed in the various coloring liquids for 2 min, rinsed with water, air-dried, and sintered. The largest surface roughnesses were observed for the test bars that were burned with dark coloring agents, VL-5 and IS-5. Figure 9 shows the SEM images of the Vickers impressions and cracks in the test bars. The marginal borders of the impressions formed using an indenter are clear and linear, and no damage, including chipping, is observed. The cracks extend in long, thin, straight needle shapes from the 4 corners of each indentation.

DISCUSSION

The indentation fracture (IF) method consists of the following processes: Vickers indenters are pressed onto a brittle solid surface with a Vickers hardness tester; semicircular or semielliptical vertical cracks are generated around the impressions; the fracture strength of ceramics is evaluated based on the size of cracks[28-32]. Although the zirconia showed clear impressions under loads of 1, 5, and 10 kgf for loading times of 15 and 30 s during the fracture toughness measurements, cracks did not occur. Clear indentation and measurable cracks occurred in the zirconia under a load of 20 kgf for a loading time of 15 s, so these conditions were used to evaluate fracture toughness. Okada *et al.*[33] evaluated the fracture toughness of dental porcelains and reported that no cracks had occurred under a load of 1 kgf for loading times of 15 and 30 s. Under a load of 5 kgf for loading times of 15 and 30 s and under a load of 10 kgf for loading times of 15 s, measurable cracks and indentations occurred in the zirconia. It was also previously reported that chippings occurred around the indentations and that cracks were not measurable under a load of 20 kgf for loading times of 15 s or longer. The Vickers indentation in Fig. 10 clearly shows a diamond-like four-sided pyramid whose diagonal plane is 136° in the zirconia. The Vickers hardness under this condition was 1,267–1,351 HV, and the coefficient of variations was 1.1–2.7%. The marked indentations, which were marked under the same conditions as for metal materials, were a normal type. The Vickers hardness of the porcelains, on the other hand, was 666–1,348 HV, and the coefficient of variations was 7–11%[34]. Chippings occurred around the porcelains, and the margins were not clearly defined. Therefore, the marked indentations were more reproducible in the zirconia than in the porcelains, and low-variability measurements could be performed for the zirconia. Cracks extended in straight lines from all corners of the four-sided pyramid in zirconia, but the cracks showed different curved forms in dental porcelains. Although the coefficient of variations for the length of cracks in zirconia was 1.0–3.4%, those for the length of cracks in the porcelains were 1.3–13.9 CV%. In comparison, the coefficient of variations for the length of cracks in zirconia was smaller. Typically, crack extension advanced after fracture and advanced from

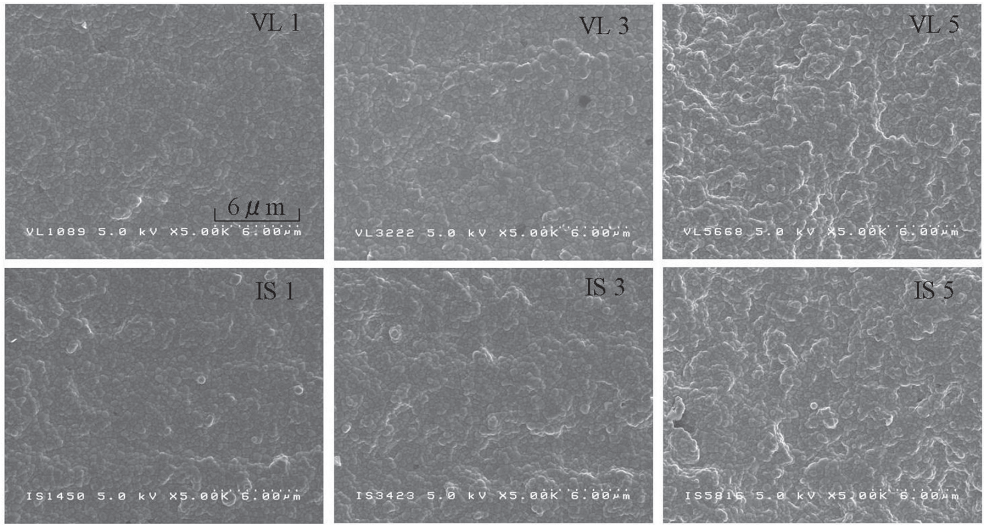

Fig. 8 SEM micrographs of fractured surface on zirconia bars after dipping bars in VL-1, 3, or 5 or in IS-1, 3, or 5.

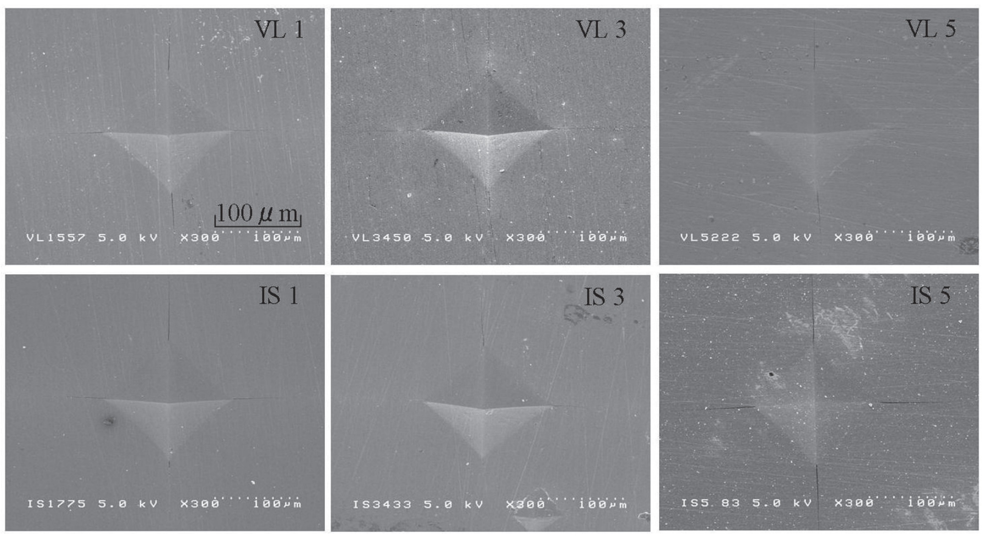

Fig. 9 SEM micrographs of indentations and cracks on zirconia bars for VL-1, 3, or 5 and for IS-1, 3, or 5.

the onset of fracturing at the notch tip to the formation of the frontal process zone to the process zone wake where the frontal process zone was expanded because of crack extension and branching and finally reached the outer surface of the edge of the sample. The higher toughness of zirconia, on the other hand, is due to stress-induced phase transition, bowed cracks, microcracking, and surface compression stress. In particular, the transformation mechanism due to stress-induced phase transition contributed significantly to the high toughness of zirconia[35]. The reason the coefficient of variations for the length of cracks in zirconia was smaller is described as follows: A tetragonal crystal stabilized at a room temperature is transformed into a monoclinic crystal under a load. Following the crystal phase transformation, a 4% cubical expansion occurs. The extension of cracks due to cubical expansion induces the crystalline phase transition from tetragonal crystal to monoclinic crystal during cubical expansion. Compression stress that occurs at the crack tip prevents the extension of cracks. In contrast, the elastic distortion energy due to an indented load on porcelain acts as a crack extension force. As a result, precipitated leucite crystals are nonuniformly distributed, which increases the coefficient of variations for the length of cracks. In addition, this force is generated by the difference in

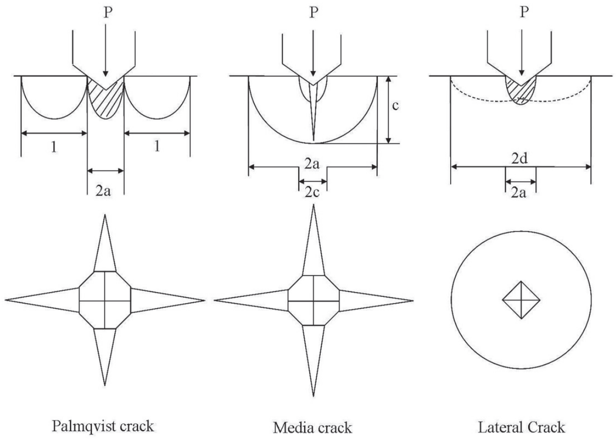

Fig. 10 Schematic illustration of forms of cracks induced by Vickers indentation on surface of zirconia.

thermal expansion between the leucite and the matrix and by the changes in volume due to the polymorphic deformation of the leucite[33]. The difference between the coefficients of variations for the length of cracks in porcelains and in zirconia is due to the difference in the previously described mechanisms. The forms of cracks generated by the Vickers impressions include Palmqvist, median, and lateral cracks, as shown in Fig. 10. The forms of cracks observed in the zirconia were either Palmqvist or median cracks because they were observed on the surface. Although cracks that occurred internally could not be observed, we determined based on our experience that the internal cracks in the zirconia were median cracks because the ratio (c/a) of the length of the crack (c) to the length of the diagonal line (a) was 2.3. When a facture face was observed during crack extension in zirconia, the cracks extended in a straight-needle-like shape. The SEM images of the surface of the dipped zirconia bars in Fig. 8 show a tetragonal polycrystalline body stabilized by 3 wt% Y_2O_3, which is consistent with the previously described mechanism of preventing crack extension. The mechanism of preventing crack extension in porcelains, on the other hand, is activated by an intergranular fracture that extends bending along a crystal grain boundary of precipitated leucite and an intergranular fracture that extends crossing in leucite crystals[36,37]. The fracture toughness of ceramics prepared with various compositions of zirconia are as follows: 3.11, 1.9, 2.78, 3.02, 4.7, 9.5, and 19.2 for Pencraft, Vitadur, Cryscera, IPS Empress 2, In-Ceram Alumina, Cercon, and NANOZR, respectively. The fracture toughness of the zirconia used for this experiment was 20.00, higher than that of any of these ceramics. We believe that this is because of the previously described mechanisms.

The optical transparency of the Y-TZP tetragonal zirconia polycrystal is low because of light scattering due to a high refractive index in the range 2.15–2.18. In addition, various shades of zirconia exhibit high luminance; more specifically, a high degree of whiteness because of the high degree of light scattering at the zirconia crystal interface. That is why the reproducibility of crown shades in clinical practice requires careful attention. In this experiment, coloring agents were applied during sintering to dye the surface of white zirconia close to the color of dentine, which enabled us to adjust the basic colors in order to produce a multilayer structure. Shades 1, 3, and 5 of Vita in-ceram 2000 YZ coloring liquid (VL) and IPS e.max ZirCAD coloring liquid (IS) were used as coloring agents. On the basis of the Vita shade guide, coloring agent shades 1, 3, and 5 corresponded to A2, B2, and D4, respectively. The bending strength of the undyed specimen was 936 MPa. Compared with that of the undyed specimen, the bending strength deteriorated in the range 12.9–33.9% for the specimens dyed with shades 1 to 5 and then sintered. The Vickers hardness of the controls was 1350. Compared with that of the controls, the Vickers hardness deteriorated in the range 0.3–5.7% for the specimens dyed with shades 1 to 5 and then sintered. The fracture toughness of the undyed specimen was 20.01. Compared with that of the undyed specimen, the fracture toughness deteriorated in the range 4.8–7.5% for the samples dyed with either IS 1 or IS 3 and then sintered to those dyed with either VL 3 or VL 5 and then sintered. The bending strength of the dyed zirconia decreased a maximum of 34%, and the fracture toughness only decreased a maximum of 8%. This extent of the effect of the coloring agents on the deterioration of the mechanical properties of zirconia is unlikely to cause problems. However, the deterioration of the mechanical properties of a dyed zirconia surface and the strength of the adhesion between the surface and layering porcelain must be examined.

Standard materials used to restore crowns and bridges have recently been changing from porcelain-fused-to-metal-based materials to all-ceramic-based ones. Restoration materials were also developed with metallic liners. All-ceramic-based materials have recently been produced using high-intensity zirconia frameworks for application in dental prosthetics. Ceramic restoration has been applied in a variety of clinical settings ranging from inlays, onlays, laminate veneers, and crowns in the early stages of tooth restoration to bridges in order to replace 1 or 2 missing teeth. The ceramics used for such restorations have been produced using the model investment, glass-infiltrated, and injection-molded methods. Zirconia frameworks recently developed using CAD/CAM systems have attracted attention as high-intensity ceramic materials that exhibit performance characteristics equal to or greater than the metal frameworks used to date for bridges. In addition, the zirconia frameworks are expected to be useful as implant abutments and superstructures[38-45]. The effect of coloring agents on the shade adjustment of zirconia frameworks was determined in order to more appropriately reproduce the shades of natural teeth. Coloring agents chemically bonded to zirconia have the same effect as an opaque layer and cause no significant deterioration of the mechanical properties of zirconia.

Therefore, we believe that clinical application can be recommended with no problem. The developed materials should enable full mouth reconstruction with totally esthetic restorations based on the application of zirconia ceramics.

CONCLUSION

This study examined the effects of coloring agents (Vita in-ceram 2000 YZ coloring liquid (VL) and IPS e.max ZirCAD (IS)) and shades (1, 3, and 5) applied during sintering on the bending strength and fracture toughness of zirconia ceramics. No differences in the bending strength or the fracture toughness of the zirconia ceramics were observed for the type of coloring agent applied. Although the bending strength and Vickers hardness of the zirconia ceramics decreased because of the various shades of coloring agents applied during sintering, the crack length and fracture toughness of the zirconia ceramics did not change. The SEM image of the zirconia ceramic dyed with dark type shade 5 and then sintered revealed that the surface of the zirconia ceramic coarsened. The marginal borders of the indentations formed using an indenter were clear and linear, and no damage, including chipping, was observed. The cracks extended in thin, long, straight, needle-shaped lines. On the basis of these results, clinical application of zirconia ceramics can be recommended with no problem because the coloring agent shades applied during sintering have the same effect as an opaque layer and cause no significant deterioration of the mechanical properties of the zirconia ceramics.

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