

Original Article

Effect of amount of grinding on flexural strength of dental ceramics

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The aim of this study was to determine a suitable amount of grinding of a flexural test specimen for clinical evaluation of dental ceramics.

Three dental ceramics (Crys-Cera, OCC and IPS Empress Cosmo) were selected. Five types of bar-shaped patterns were prepared for a three-point bending test, and four types of disk-shaped patterns for a biaxial flexural test. Ceramic specimens were fabricated using these patterns in accordance with the manufacturers' instructions. All specimens were ground with diamond polishing pads to yield either bar or disk specimens with the same final geometry. The surface roughness and X-ray diffraction (XRD) patterns of the specimens were examined.

The flexural strength values of Crys-Cera and Empress decreased with increasing amounts of grinding, while that of OCC increased with increased grinding. The surfaces of all specimens were smoother when the amount of grinding was deeper. The XRD patterns of Crys-Cera and Empress changed with increased grinding; however, that of OCC did not change regardless of the extent of grinding.

The flexural strengths of three dental ceramics differed with the amount of grinding probably because of changes of surface roughness and crystalline component composition. Unground specimens provided important insight into clinical failure modes.

Key words: Dental ceramics; Flexural strength; Grinding; Three-point bending test; Biaxial flexural test

Introduction

Ceramics applications in the dental field have increased because of patients' demands for aesthetic restoratives^{1,2} and problems due to metal allergy³. Ceramics have been utilized for inlays and crowns and also for post and core systems^{4,5}. Newly introduced dental ceramics have improved mechanical properties according to their manufacturers.

Mechanical properties of dental ceramics are usually evaluated by a flexural test⁶. The ISO specification for dental ceramics⁷ requires 0.4 mm grinding of each surface of the ceramic specimen for dimensional preparation for the flexural test; however, the inner side of a restoration adjacent to the tooth is not usually ground in such a manner and ceramic fractures often occur from this side⁸. Moreover, crystalline components of some castable ceramics were considered to differ between surface and bulk because of their crystallized mechanism^{9,10}. In this study, the effect of the amount of

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grinding of ceramics on its mechanical strength was evaluated in an effort to determine suitable specimen preparation conditions for the evaluation of mechanical properties of dental ceramics.

Materials and Methods

Materials

Three dental ceramics were selected for this study: two castable ceramics (Crys-Cera, Kyutai Dentceram, Okayama, Japan; OCC, Olympus Optical, Tokyo, Japan) and one hot-pressed ceramic (IPS Empress Cosmo, Ivoclar, Schaan, Liechtenstein) (Table I). Crys-Cera is a calcium phosphate castable ceramic; the crystallization procedure is performed in a special investment. The crystallization occurs from the interface of the investment⁹. OCC is a mica glass castable ceramic; the crystallization procedure occurs randomly from inside the ceramic¹¹. IPS Empress Cosmo is a hot-pressable ceramic containing zirconia designed for the post and core systems in combination with of a zirconia prefabricated post¹².

Preparation of the specimen

The specimens were prepared with and without

grinding. Five types of bar-shaped patterns (SA: 5.0 mmW × 20.0 mmL × 2.0 mmD, SB: 5.0 mmW × 20.0 mmL × 1.6 mmD, SC: 5.0 mmW × 20.0 mmL × 1.4 mmD, SZ: 5.0 mmW × 20.0 mmL × 1.2 mmD, SZZ: 4.0 mmW × 20.0 mmL × 1.2 mmD) for a three-point bending test and four types of disk-shaped patterns (RA: 14.0 mm diameter (ϕ) × 2.0 mmD, RB: 14.0 mm ϕ × 1.6 mmD, RC: 14.0 mm ϕ × 1.4 mmD, RZ: 14.0 mm ϕ × 1.2 mmD) for a biaxial flexural test were prepared (Table II).

All patterns except for SZ and SZZ were made from wax using brass molds. For producing sharp edges of the patterns, the patterns for SZ and SZZ were made from an acrylic plate by grinding with a #1000 diamond polishing pad (Maruto, Tokyo, Japan) with a precision grinding machine (Dia-Lap ML-150P, Maruto, Tokyo, Japan).

Ceramic specimens were fabricated using these patterns in accordance with the manufacturers' instructions. Heating and casting conditions are shown in Table III. Sandblasting with glass beads (diameter of 50 μ m) devested the specimens, and crystallization of Crys-Cera and OCC was done. All specimens except SZZ and RZ were ground with diamond polishing pads (#600 and #1000) to form the same geometry of SZZ or RZ using the precision grinding machine with running water under a load of 9.8 N at a

Table I. Materials used in this study

Product	Type of ceramics	Manufacturer	Batch No.
Crys-Cera	castable / CaO-P ₂ O ₅ -Al ₂ O ₃	Kyutai Dentceram, Okayama, Japan	980128 No.1.5
OCC	castable / Li ₂ O-MgO-ZnO-TiO ₂ -Al ₂ O ₃ -SiO ₂	Olympus Optical, Tokyo, Japan	OCS-2P-98-2-002M
IPS Empress Cosmo	pressable / SiO ₂ -ZrO ₂ -Li ₂ O-P ₂ O ₅	Ivoclar, Schaan, Liechtenstein	A05150

Table II. Dimensions of wax patterns used

Code	3-point bending test	Code	Biaxial flexural test
SZZ	4.0W X20.0L X1.2D		
SZ	5.0W X20.0L X1.2D	RZ	14.0 ϕ X1.2D
SC	5.0W X20.0L X1.4D	RC	14.0 ϕ X1.4D
SB	5.0W X20.0L X1.6D	RB	14.0 ϕ X1.6D
SA	5.0W X20.0L X2.0D	RA	14.0 ϕ X2.0D

W: width, L: length, D: thickness, ϕ : diameter

Unit:mm

Table III. Condition for casting and ceraming

Program	Crys-Cera	OCC	IPS Empress Cosmo
Heating rate (°C/min)	5	30	5
Maximum temp. of mold (°C)	800	800	850
duration time (min)	30	40	10
Holding temp. of mold (°C)	530	520	-
Hot press temp. (°C)	-	-	700
Casting temp. of ingot (°C)	1310	1280	-
Heat press temp. of ingot (°C)	-	-	900
Ceraming temp. (°C)	640	883	-
duration time(hrs)	2	0	-

rotational speed of 85 revolutions per minute. Half of the prescribed grinding amount employed the #600 diamond pad and the rest, the #1000 pad. The grinding amount of the upper surface and that of the lower surface were checked to be uniform. Specimens of SZZ and RZ were employed without grinding. The sizes of grinding of all specimens were measured using a micrometer (CPM15-25DM, Mitsutoyo, Tokyo, Japan) with 0.001mm precision. The final dimensions of the specimens were within a 0.01 mm range of the prescribed size.

Flexural property

Flexural properties of the specimens after 24 h distilled water storage at 23°C were examined by three-point bending and biaxial flexural tests in basic accordance with ISO 6872:1995. The dimension of each specimen was measured to 0.01 mm before testing. A universal testing machine (1123, Instron, Canton, MA, USA) was employed at a crosshead speed 1.0 mm/min. The three-point bending test was performed in air at room temperature with a 14.0 mm support span; the biaxial flexural test was done in distilled water at room temperature with a 10.0 mm support circle and a 1.2 mm piston diameter. Ten specimens were examined for each test. The three-point bending strength and flexural elastic modulus were calculated using analyzing software (Series IX, Instron). The flexural strength was calculated by the following equation:

$$M = (3WI) / (2bd^2)$$

where

- M is the flexural strength, in megapascals;
- W is the breaking load, in newtons;
- l is the test span (centre-to-centre distance between supports), in millimetres;
- b is the width of the specimen, i.e. the dimension of the side at right angles to the direction of the applied load, in millimetres.
- d is the thickness of the specimen, i.e. the dimension of the side parallel to the direction of the applied load, in millimetres.

The biaxial flexural strength was calculated by the following equation:

$$S = -0.2387P(X-Y)/d^2$$

where

- S is the maximum centre tensile stress, in megapascals;
- P is the total load causing fracture, in newtons;

$$X = (1+\nu)\ln(r_2/r_3)^2 + [(1-\nu)/2](r_2/r_3)^2$$

$$Y = (1+\nu)[1+\ln(r_1/r_3)^2] + (1-\nu)(r_1/r_3)^2$$

in which

- ν is Poisson's ratio, the value for this study was 0.25;
- r_1 is the radius of support circle, in millimetres;
- r_2 is the radius of loaded area, in millimetres;
- r_3 is the radius of specimen, in millimetres;
- d is the specimen thickness at fracture origin, in millimetres.

Obtained properties were statistically analyzed using the one-way analysis of variance and Scheffe's multiple comparison test.

Surface roughness

Surface roughness of all specimens after the three-point bending test was analyzed using a surface roughness analyzer (Surfcom, 50A, Tokyo Seimitsu, Tokyo, Japan). Three measurements were performed for each specimen according to ISO 4287:1997¹³. The maximum height (Rz) was measured with a cut-off value of 0.8, a measuring length of 2.0 mm, and a measuring speed of 0.3 mm/min. Obtained Rzs were analyzed using one way analysis of variance and multiple comparison.

SEM observation

The surfaces of three ceramic specimens of SA, SB, SC and SZ coated with carbon evaporation were observed using a scanning electron microscope (DS130C, Akashi Beam Technology, Tokyo, Japan).

X-ray diffraction (XRD)

The crystalline components of the specimens were analyzed using an X-ray diffractometer (RAD-II A, Rigaku, Tokyo, Japan) with Cu-alpha radiation of 40 kV and 20mA and a 0.5° /min scanning speed. The XRD patterns of the RZ specimen without grinding were obtained; those of the RZ specimen after a 0.1, 0.2, 0.3, and 0.4 mm grinding with the #1000 diamond pad were sequentially obtained. The crystalline components of the ceramic surface were identified using an XRD pattern processing and identification software (MDI JADE 4.0, Materials Data, Livermore, CA, USA).

Results

Flexural property

Flexural strengths and standard deviations obtained by the three-point bending and the biaxial flexural tests are shown in Fig. 1 and Fig. 2, respectively. The bars connected with a horizontal line were not different at a 0.05 significance level. The flexural strengths of Crys-Cera and Empress decreased with

increasing amounts of grinding; however, those of OCC had a tendency to increase with increased grinding.

With respect to Crys-Cera, both the three-point bending and the biaxial flexural strengths varied significantly with the amount of grinding. The strength of SZZ, 201.2 MPa, was significantly greater than those of the other groups ($p < 0.05$), SZ, SC, SB, and SA, which were not significantly different from each other. The biaxial flexural strength of RZ, 176.1 MPa, was significantly greater than those of the other groups; the biaxial flexural strengths of RC, RB, and RA were not significantly different from each other.

Concerning OCC, the three-point bending strength of SZZ, 202.0 MPa, was the smallest value among the other groups; however, there was no significant difference among all groups. Similar results were obtained for biaxial flexural strength. The biaxial flexural strength of RC, 187.9 MPa, was the smallest value among the other groups; there was no significant difference among all groups.

Regarding Empress Cosmo, the three-point bending strength and the biaxial flexural strength were varied significantly with the amount of grinding; the three-point bending strength of SZZ, 142.8 MPa, was significantly greater than those of SA, SB, and SC, but the same as SZ. The biaxial flexural strength of RZ, 117.6 MPa, was significantly greater than those of the other groups; the biaxial flexural strengths of RC, RB, and RA were not significantly different from each other.

Elastic moduli of Crys-Cera ranged from 51.4-58.9 GPa; those of OCC were 49.9-60.1 GPa; those of Empress 58.0-72.2 GPa. There was no obvious relation between the elastic modulus and the amount of grinding.

Surface roughness

The results of maximum height (Rz) and standard deviation are shown in Fig. 3. The surface roughness of all ceramics revealed that the surfaces of the specimens were smoother when the grinding depth was increased. Rzs of SZ and SZZ, as well as those of SA and SB, were not significantly different regardless of the types of the ceramics. With respect to OCC, Rzs of SA, SB and SC were not significantly different.

SEM observation

The surface of the three ceramics without grinding showed similar images; the surface images of RZ were rough and irregular. The surface images of the ceramics after grinding were obviously different from

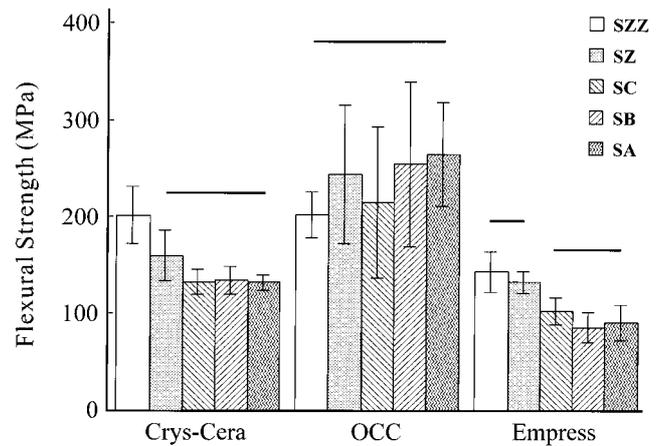


Fig. 1. Flexural strength of the three ceramics tested in air. The bars connected with a horizontal line were not significantly different.

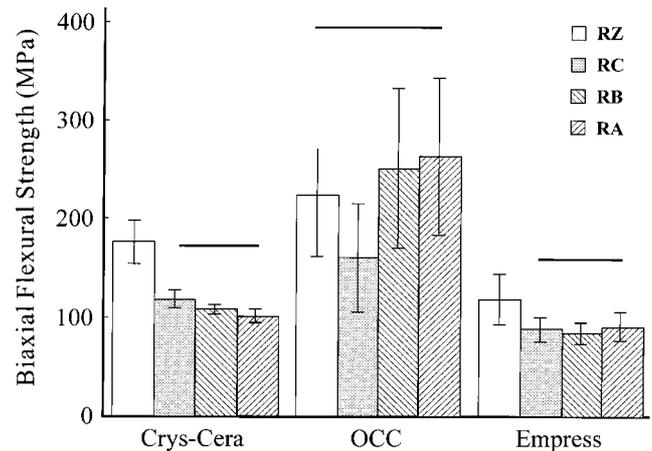


Fig. 2. Biaxial flexural strength of the three ceramics tested in water. The bars connected with a horizontal line were not significantly different.

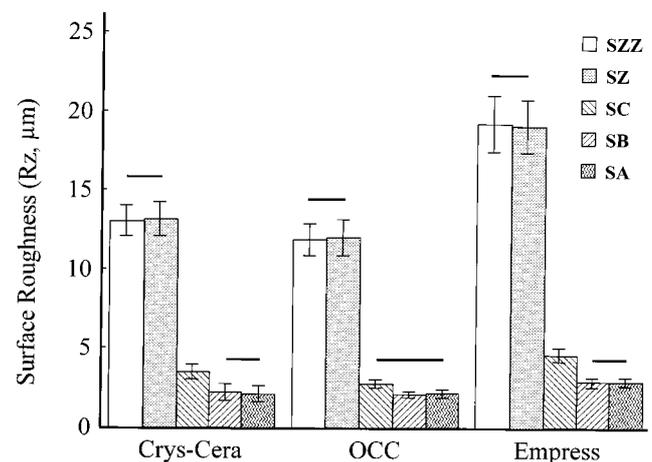


Fig. 3. Surface roughness (Rz) of the specimens for the three-point bending test in air. The bars connected with a horizontal line were not significantly different.

those without grinding. After grinding, a great number of grinding scratches at random directions were observed on the surfaces of RC, RB, and RA. However, there were no obvious differences among RC, RB and RA. The surface of the Empress after grinding was different from those of the other two types of ceramics because of the existence of some spherical porosity (Fig. 4).

X-ray diffraction

The XRD patterns without grinding and after 0.1 mm, 0.2 mm, 0.3 mm and 0.4 mm grinding from the surface of the specimens are shown in Fig. 5, 6, and 7.

Regarding Crys-Cera and Empress, the XRD patterns before and after grinding were obviously different. Crystalline components of Crys-Cera consisted of LiCaP_3O_9 , CaP_2O_6 and NaAlSiO_4 . By grinding the specimens, the peaks assigned to LiCaP_3O_9 became smaller, whereas those of CaP_2O_6 increased. Crystalline components of Empress consisted of

ZrSiO_4 , Li_3PO_4 and SiO_2 . By grinding the specimens, the peaks assigned to SiO_2 became smaller, whereas those of ZrSiO_4 increased. However, the XRD patterns of OCC before and after grinding were almost identical. Crystalline components of OCC were considered $\text{LiAl}(\text{SiO}_3)_2$ and $\text{NaMg}_3\text{AlSi}_3\text{O}_{10}\text{F}_2$.

Discussion

A flexural test is usually employed for the evaluation of dental materials because it is relatively easy to prepare specimens. The three-point bending test and the biaxial flexural test are suggested in ISO6872: 1995 for dental ceramics. In this study, the three-point bending test was performed in air while the biaxial flexural test was performed in water. Considering intraoral environment, the flexural test should be performed in water. However, the flexural strength of ceramics in water is reported to be smaller than that in air¹⁴. We measured

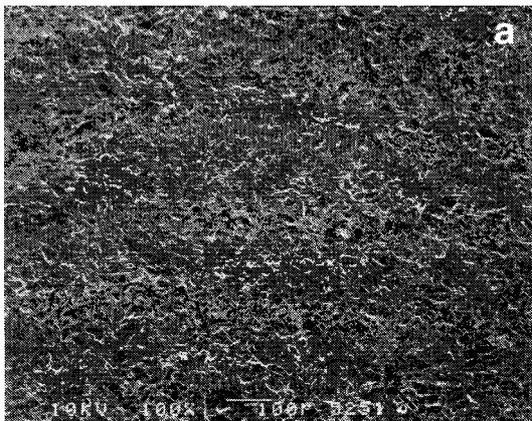


Fig. 4. SEM images of ceramic surface without grinding (a) and after 0.4 mm grinding (b) (Empress Cosmo).

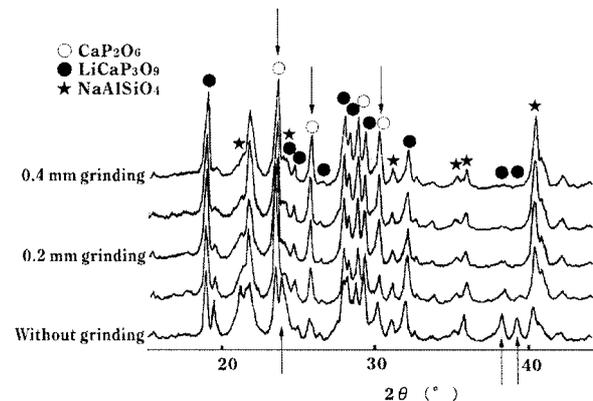


Fig. 5. Changes of X-ray diffraction pattern (Crys-Cera). The upward arrows show the decreasing peaks by grinding; on the other hand, the downward arrows show the increase peaks by grinding.

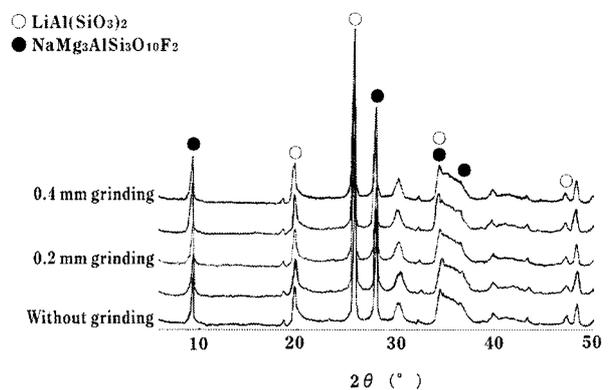


Fig. 6. Changes of X-ray diffraction pattern (OCC).

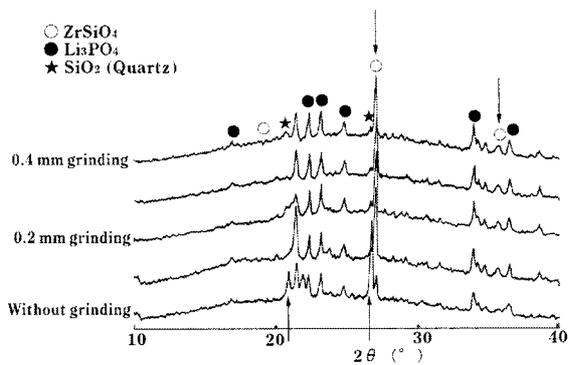


Fig 7. Changes of X-ray diffraction pattern (Empress Cosmo). The upward arrows show the decreasing peaks by grinding; on the other hand, the downward arrows show the increase peaks by grinding.

the biaxial flexural strength of Crys-Cera in air according to the same manner in this study; the ratios of the biaxial flexural strength of RZ, RC, RB and RA in water to that in air were 0.709, 0.761, 0.644 and 0.665, respectively. Thus, caution should be used when comparing the strength in water and that in air. Moreover, the edge of a castable ceramic restoration is usually not rounded in laboratory procedures. Therefore, in this study, the edges of the specimens were not rounded. As a result, the flexural strengths of the unground “as cast” specimens influenced by the sharp edge reproducibility of castable ceramics.

The three-point bending strengths of Crys-Cera, OCC and Empress Cosmo were reported 116 MPa¹⁵, 220-300 MPa¹¹ and 164 MPa¹², respectively. Three-point bending strengths of Crys-Cera and OCC were almost identical to those reported values; however, that of Empress Cosmo was smaller than the reported value in spite of following the manufacturer's instructions.

It is reported that there is a low mechanical strength layer on the surface of a certain castable ceramics¹⁰. Moreover, the surface layer of the castable or hot-pressable ceramics might react with the mold component and be stressed by cooling during the process of fabrication. In addition, the surfaces of ceramic specimens are usually very rough, and the strength of the ceramics improves when its surface becomes smooth¹⁶. Therefore, the relatively great amount of grinding of ceramics might be suggested in the specification. The ground surface of specimens in this study showed fine scratches at random directions. The relationship between the amount of grinding and the flexural strength is shown in Fig. 8. When the surface roughness decreased by polishing, the flexural strength of OCC was reported to increase¹⁷, while that

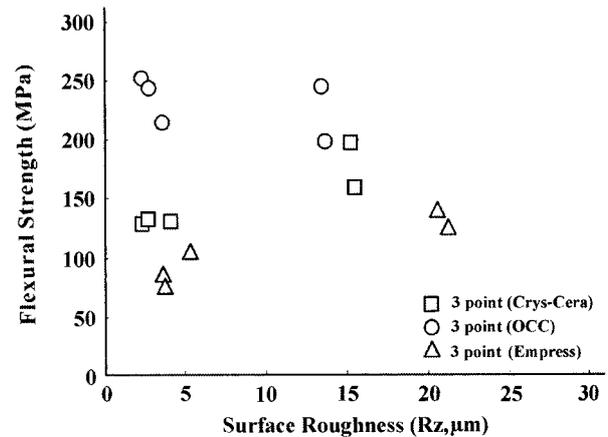


Fig 8. Relationship between the surface roughness and the flexural strength.

of Crys-Cera decreased. Therefore, the key of flexural strength of ceramics is not only the surface roughness. Crushed powders of Crys-Cera and Empress obtained from the unground surface were also examined; the XRD patterns of the powders were not identical to those of the unground specimen surface. These results suggest that anisotropic crystalline orientation due to casting and the crystallization procedure could complicate the XRD patterns. Therefore, several peaks contained in the XRD patterns were not identified. However, the XRD patterns of Crys-Cera and Empress before grinding were not identical to those after grinding. Crystallization of OCC occurs from the entire glass ceramic body¹¹; on the other hand, that of Crys-Cera is from its surface⁹. As a result, the crystalline compositions of the surface portion of Crys-Cera were different from those of the internal portion. This finding might be another key to the strength of dental ceramics.

The origins of clinical failure of dental ceramics have been reported to be at the load-applied point and its internal surface¹⁸; generally, the inner surface of the casting is usually “as cast” and not polished. Therefore, a specimen without grinding might be more desirable for clinical fracture evaluation.

On the other hand, defects and flaws on the edges of the tensile side surface of the three-point bending test specimen have a great influence on the flexural strength, while those of the biaxial flexural test specimen do not. Therefore, flexural strength by the biaxial flexural test using the specimen without grinding might be suitable for predicting clinical performance of dental ceramics. However, the biaxial flexural test only provides a flexural strength value. As a result, the

three-point and four-point bending tests are still useful for obtaining the other mechanical properties such as the flexural modulus and toughness.

For clinical application of dental ceramic restorations, the surface of the restorations are often ground or polished because of adjustment for occlusal or proximal contacts. However, such kind of grinding or polishing affects the strength of the dental castable ceramic restoration. Therefore, it is necessary to fabricate dental ceramic restorations more accurately than metallic restorations to avoid restoration fractures due to additional grinding or polishing before cementation.

Conclusion

Flexural strengths of three types of dental ceramics differed with the amount of grinding most likely because of changes in the surface roughness and in the crystalline component composition. Regarding the clinical fracture situation, a specimen without grinding might be useful for predicting clinical performance of dental ceramics.

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