Chemical Solubility and Flexural Strength of Zirconia-Based Ceramics

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> Purpose: This study was undertaken to investigate the chemical solubility and flexural strengths of 3 zirconia-based dental ceramics: In-Ceram Zirconia (IZ), In-Ceram 2000 YZ CUBES (YZ Zirconia) (Vita Zahnfabrik), and Cercon (Dentsply). A pressable lithium disilicate-reinforced glass ceramic (IPS Empress 2, Ivoclar Vivadent) was used as a control. *Materials and Methods:* Ten block specimens $(12 \times 6 \times 4 \text{ mm})$ of each ceramic material were prepared for chemical solubility testing. Each specimen was refluxed in 4% acetic acid solution for 16 hours. The percentage loss of mass and the loss of mass per unit of surface area for each specimen were calculated. Ten barshaped ($21 \times 5 \times 2$ mm) and 10 disk-shaped (16 mm diameter, 1.2 mm thickness) specimens of each ceramic material were prepared and tested for uniaxial flexural strength (UFS) and biaxial flexural strength (BFS). X-ray diffraction analyses were conducted to determine the relative amount of the monoclinic phase of the as-sintered and fractured surfaces of the zirconia ceramics. Results: There were no significant differences among the ceramics in chemical solubility by percentage mass or mass/surface area. For UFS, YZ Zirconia (899 ± 109 MPa) > Cercon (458 ± 95 MPa) IZ (409 ± 60 MPa) > Empress 2 (252 ± 36 MPa). For BFS, YZ Zirconia (1,107 ± 116 MPa) > Cercon (927 ± 146 MPa) > IZ (523 ± 51 MPa) > Empress 2 (359 ± 43 MPa). The fractured YZ Zirconia surface contained approximately 5 times as much monoclinic phase compared to that of its intact surface. The fractured IZ and Cercon surfaces contained approximately twice as much monoclinic phase compared to those of intact surfaces. Conclusion: The ceramics tested all satisfied the chemical solubility allowance required of core ceramic material (type I, Class 1 or type II, Class 1) according to the International Organization for Standardization 6872:1995(E) specifications on dental ceramic. The zirconia-based ceramics possessed significantly higher flexural strengths than the control lithium disilicate ceramic. Their clinical application appears sufficiently promising for long-term clinical studies to be undertaken. Int J Prosthodont 2007;20:587-595.

Zirconia-based ceramics are the latest introduction of exceptionally high-strength materials to dentistry. The strength and toughness of zirconia-based ceramic can be accounted for by its toughening mechanisms, such as crack deflection, zone shielding, contact shielding, and crack bridging.^{1,2} The most important among the toughening mechanisms appears to be contact shielding mediated by the tetragonal to monoclinic phase transformation. Under applied stress, the tetragonal phase undergoes phase transformation into the monoclinic phase, which is 4% larger in volume. This volume increase induces compressive stress in the vicinity of the crack tip, thereby constraining growth. It is also possible for phase transformation to occur when the ceramic is ground or polished.^{3–6}

In addition to mechanical properties, the chemical solubility of a dental ceramic is an important criterion for material selection. Exposure to the various acidity

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Table I Ceramic Materials Used in the Study	Table 1	Ceramic I	Materials	Used	in the	Study
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Material	Manufacturer	Major component
In-Ceram Zirconia	Vita Zahnfabrik	Zirconia-reinforced, glass-infiltrated alumina ceramic
In-Ceram 2000 YZ CUBES	Vita Zahnfabrik	Yttria-stabilized zirconia ceramic
Cercon	Dentsply	Yttria-stabilized zirconia ceramic
IPS Empress 2	Ivoclar Vivadent	Lithium disilicate-reinforced glass ceramic

of foodstuffs may cause dissolution of a ceramic material, which in turn may cause a loss of surface luster, an increase in plaque retention, or weakening of the material.^{6,7}

One example of zirconia-based ceramic is In-Ceram Zirconia (IZ) (Vita Zahnfabrik). Based on the concept of In-Ceram Alumina (IA), IZ was developed with the addition of 33% 12 mol% ceramic oxide (CeO₂) partially stabilized zirconia to IA.³ The two varieties of IZ, slip or dry-pressed millable materials, require glass infiltration to develop full strength. The flexural strengths of slip and dry-pressed IZ were found to be 630 and 476 MPa, respectively.³ The superiority in strength of IZ over IA was reported in 3 studies,^{4,8,9} but was not affirmed in 1 study.¹⁰

In-Ceram 2000 YZ CUBES (YZ Zirconia) (Vita Zahnfabrik) is a newly developed yttria-stabilized zirconia ceramic containing approximately 95% zirconium oxide (ZrO_2) and 5% yttrium oxide (Y_2O_3). The material is marketed as presintered zirconia blanks used together with a computer-aided design/computer-assisted manufacture (CAD/CAM) system (Cerec 3/InLab, Sirona Dental Systems). The milled presintered ceramic blank is then fired in a high-temperature furnace to complete sintering.

Cercon (Dentsply) is a 3 mol% yttria-stabilized zirconia ceramic that employs its own CAM system in milling a presintered zirconia blank according to the scanned wax pattern of the ceramic core. Similar to YZ Zirconia, the milled ceramic blank is further sintered to complete fabrication.¹¹

Although the strength of yttria-stabilized zirconia ceramics is well documented,^{3,12-15} data on the properties of zirconia-based dental ceramics are not readily available. Thus, the purpose of the study was to investigate the chemical solubility and uniaxial flexural strength (UFS) and biaxial flexural strength (BFS) of 3 zirconia-based dental ceramics.¹⁶ A pressable lithium disilicate-reinforced glass ceramic was used as the control.

Materials and Methods

Three zirconia-based dental ceramic materials were studied together with 1 lithium disilicate ceramic (IPS Empress 2, Ivoclar Vivadent), which acted as the control (Table 1). An international standard for the testing of dental ceramic, International Organization for Standardization (ISO) 6872:1995(E), was adopted for the experiment.¹⁶

In the present study, IZ specimens (bars and disks) were prepared using the molds of dimensions specified by ISO 6872:1995(E). The IZ slip in a special plaster cast was trimmed to the desired dimensions using sharp scalpels before sintering and glass infiltration. After removal of excess glass by sandblasting, finishing was performed using 600-grit silicon carbide paper.

For the Cercon specimens, the bars and disks were fabricated by scanning the wax patterns, milling the enlarged blanks according to the amount of linear shrinkage, and sintering the milled specimens. The specimens were finished in the same manner as the IZ specimens. The YZ Zirconia specimens were also fabricated using the computer-assisted scanning, milling, and sintering procedures. Before final polishing using the 600-grit silicon carbide paper, the sintered YZ Zirconia specimens were examined under \times 20 magnification to ensure that there were no scratches. Cutting or grinding of the sintered specimens was demanding because of their extreme hardness. Therefore, every effort was made to approximate the final dimensions of the specimens during preparation to ensure minimal surface finishing, using the 600-grit silicon carbide paper for all specimens.

Chemical Solubility

Ten specimens of each ceramic material were prepared according to the manufacturers' instructions into ceramic bars with dimensions of length (I) 12 ± 0.1 mm, width (w) 6 \pm 0.1 mm, and thickness (t) 4 \pm 0.1 mm. The surfaces of each specimen were polished with 600-grit silicon carbide abrasive paper. The dimensions of each specimen were measured to the nearest 0.05 mm with an electronic digital micrometer. A reflux-condenser-type 3-piece extraction apparatus (Pyrex, Corning Scientific Products) was used. The specimens were washed with ISO 3696 grade 3 water and dried before extraction.¹⁷ Each specimen was placed separately in a glass-bottomed thimble. Each specimen in its thimble was conditioned to constant weight by storing it at $150 \pm 5^{\circ}$ C for 4 hours in a furnace (Precision Scientific). The samples were weighed

with an electronic balance (Mettler Instrument). After further conditioning in a furnace at $150 \pm 5^{\circ}$ C, the samples were repeatedly weighed every hour within a 24-hour period until 3 readings obtained were within 0.1 mg. This value was recorded as W₁.

The thimble and specimen were then placed in the extraction apparatus, and the specimen was extracted using 4% acetic acid solution (Panreac Quimica SA, 99.7% Riqueza minima) by refluxing for 16 hours with an 18-minute cycle reflux rate. Each specimen was washed in the thimble with ISO 3696 grade 3 water.¹⁷ The thimble and specimen were conditioned again to constant weight to the nearest 0.1 mg at 150 \pm 5°C within a 24-hour weighing cycle. Weighing was repeated and the value was noted as W₂.

The percentage loss of mass for each specimen was calculated using the formula:

$$(W_1 - W_2) / W_1 \times 100\%$$

The loss of mass for each specimen based on surface area was calculated using the formula:

$$(W_1 - W_2) / 2 (I \times w + I \times t + w \times t)$$

One-way analysis of variance (ANOVA) and Neuman-Keuls post hoc tests were applied to determine any significant differences between the specimens (P=.05)

Uniaxial Flexural Strength (3-Point Bend Test)

An international standard for the testing of dental ceramic, ISO 6872:1995(E), was adopted for the experiment.¹⁶ Ten bar-shaped specimens of each ceramic material ($21 \times 5 \times 2$ mm) were prepared. IZ, Cercon, and IPS Empress 2 specimens were prepared in accordance with the manufacturers' instructions. YZ Zirconia specimens were provided by the manufacturer. The rectangular specimens were ground to maintain parallel surfaces to a width, thickness, and length of 4.0 \pm 0.25 mm, 1.2 \pm 0.2 mm, and 21 \pm 0.2 mm, respectively. All specimens were finished with 600-grit silicon carbide abrasive paper, which is equivalent to a particle size of 15 µm. The dimensions were confirmed by measurement with an electronic digital micrometer. Each specimen was then cleaned thoroughly in running tap water to remove all traces of debris.

A universal testing machine (Model 1185, Instron) with a 3-point bend test jig was used to determine the UFS. Two hardened steel knife edges with a radius of 0.8 ± 0.01 mm formed the supports for the specimen. The span between the supports was 13.5 ± 0.01 mm. The midpoint of each specimen was located and then placed centrally between the supports. The load at a

crosshead speed of 1.0 mm/min was applied to the midpoint between the supports by means of a third steel knife edge with a radius of 0.8 ± 0.01 mm across the 4-mm-wide face along a line perpendicular to the long axis of the bar. The load required to fracture the test piece was measured to the nearest 0.1 N. The cross-sectional dimensions (width and thickness) of each specimen were measured to the nearest 0.01 mm at the point where the fracture occurred.

The UFS (M) of each specimen was calculated using the formula:

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

where W = the breaking load (N); I = the test span (center-to-center between supports) (mm); b = the width of the specimen (mm), ie, the dimension of the side at right angles to the direction of the applied load; and d = the thickness of the specimen, ie, the dimension of the side parallel to the direction of the applied load.

The data were subjected to 1-way ANOVA and Neuman-Keuls post hoc tests to determine any significant differences among the groups at P = .05.

Biaxial Flexural Strength Test (Piston-on-3-Ball Test)

Ten disk-shaped specimens of each material were fabricated with dimensions of 16 ± 0.1 mm in diameter and 1.2 mm in thickness (with the exception of YZ Zirconia, which was 12 ± 0.1 mm in diameter). IZ, Cercon, and IPS Empress 2 specimens were prepared in accordance with the manufacturers' instructions. YZ Zirconia specimens were provided by the manufacturer. The surfaces of the specimens were finished with 600-grit silicon carbide abrasive paper, which is equivalent to a particle size of 15 µm, until the opposing facets of the test pieces were all flat and parallel within ± 0.05 mm. The dimensions were confirmed by measurement with an electronic digital micrometer.

A piston-on-3-ball test was set up for the experiment (Fig 1). Three hardened steel balls with a diameter of 3.2 ± 0.01 mm were positioned 120 degrees apart on a support circle with a diameter of 10 mm. Each specimen was placed concentrically on the supporting balls of the testing machine so as to ensure that the load was applied at the center of the test piece. The load was applied with a universal testing machine (Model 1185, Instron) through a flat punch with diameter of 1.4 ± 0.01 mm at the center of the specimen at a crosshead speed of 1.0 mm/min. The load required to fracture each specimen was recorded to the nearest 0.1 N. The specimen thickness at fracture origin was measured to the nearest 0.01 mm.



Fig 1 Piston-on-3-ball setup for the biaxial flexural strength test.

The BFS (S) for each specimen was calculated using the formula¹⁶:

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

where X = $(1 + \nu)\ln(r_2/r_3)^2 + [(1 - \nu)/2](r_2/r_3)^2$; Y = $(1 + \nu)[1 + \ln(r_1/r_3)^2] + (1 - \nu)(r_1/r_3)^2$; P = load at failure (N); d = specimen thickness at fracture origin (mm); r_1 = radius of supporting circle (mm); r_2 = radius of loaded area (mm); r_3 = radius of specimen (mm); and ν (Poisson ratio) = 0.25.

The strength results obtained were tested using 1way ANOVA and Newman-Keuls multiple comparison test to determine any significant differences among the groups at P = .05.

X-ray Diffraction

X-ray diffraction analyses (Philips PW1830 Powder Xray Diffraction System) were conducted to determine the relative amount of the monoclinic phase of the assintered and fractured surfaces of the zirconia ceramics (IZ, YZ Zirconia, and Cercon). The intact and fractured surfaces of one randomly chosen bar (UFS test) and disk (BFS test) specimen were scanned with copper K α x-rays from 20 to 60 20 degrees with a step size of 0.05 degrees and 2-second step interval.

The relative amount (X_M) of the monoclinic phase was calculated based on the method of Garvie and Nicholson¹⁸ as:

$$X_{m} = (I_{m1} + I_{m2}) / (I_{m1} + I_{m2} + I_{t})$$

where I = the intensity detected by the detector at angular position 2 θ degrees from the diffraction; t = the tetragonal peak (20 at 30.167 degrees); m1 (\overline{I} , I, I) 20 at 28.174 degrees) and m2 (\overline{I} , I, I) (20 at 31.467 degrees) = the 2 major monoclinic peaks with reference to standard patterns archived in the PCPDFWIN software database of the International Centre for Diffraction Data (ICDD): 37-1484 for monoclinic zirconium oxide and 17-0923 for tetragonal zirconium oxide.

Microscopy

Two representative specimens of each ceramic material were randomly selected for viewing (1 unetched and 1 etched) under scanning electron microscopy (SEM) (XL30CP, Philips Electron Optics; Leica-Cambridge S440 Scanning Electron Microscope using backscatter function) to study their fracture surfaces. Unetched specimens were sputtered with gold and viewed under magnification ×4,000 and ×7,000, respectively. Zirconia-based specimens were etched by boiling in 70% sulfuric acid for 5 minutes and then left covered in a fume cupboard for drying before sputtering and viewing under magnification $\times 2,000$. An Empress 2 specimen was etched with 10 vol% hydrofluoric acid for 20 minutes, sputtered, and viewed under magnification ×2,085 to study its lithium disilicate structure.2

Results

There were no significant differences among the ceramics in chemical solubility by percentage mass or mass/surface area (Table 2). All ceramics exhibited a mean chemical solubility by mass below one-hundredth of one percent.

One-way ANOVA showed that the UFS and BFS of the ceramics were significantly different from each other. Newman-Keuls multiple-comparison tests revealed that YZ Zirconia possessed significantly higher UFS and BFS than the other ceramics (Table 2). Empress 2 had significantly lower UFS and BFS than the other ceramics. Cercon and IZ were intermediate in UFS and BFS; the UFS values of these 2 ceramics were not significantly different from each other, while the BFS of Cercon was significantly higher than that of IZ (Table 2).

The relative amounts of the monoclinic phase presenting on the intact and fractured surfaces of the disk (BFS test) and bar (UFS test) specimens of the zirconia-based ceramics are shown in Table 3. An example of the relative intensity of tetragonal and monoclinic phases of a disk specimen of YZ Zirconia is shown in Fig 2. The magnitude of change in the relative amount of monoclinic phase on the intact surface versus that on the fracture surface is comparable between the disk and bar specimens of the same ceramics. The frac-

Vaterial	Chemical solubility (%)	Chemical solubility (µg/cm²)	Uniaxial flexural strength (MPa)	Biaxial flexural strength (MPa)
n-Ceram Zirconia	$3.7 imes10^{-3}\pm5.4 imes10^{-3}$	320 ± 474	409 ± 60	523 ± 51
n-Ceram 2000 YZ CUBES	$6.1 imes10^{-3}\pm8.3 imes10^{-3}$	516 ± 704	899 ± 109	$1,107 \pm 116$
Cercon	$4.7 imes 10^{-4} \pm 4.3 imes 10^{-4}$	39 ± 35	458 ± 95	927 ± 146
PS Empress 2	$1.9 imes10^{-3}\pm2.2 imes10^{-3}$	178 ± 203	252 ± 36	359 ± 43

Table 2 Chemical Solubility and Flexural Strength of Zirconia-Based Ceramics (Means \pm SDs)

Table 3Relative Amounts (%) of the Monoclinic Phase on the Intact and FracturedSurfaces of 1 Randomly Chosen Bar and Disk Specimen for Each Zirconia-BasedCeramic

	In-Ceram Zirconia		In-Ceram 2000 YZ CUBES		Cercon	
	Bar	Disk	Bar	Disk	Bar	Disk
Intact surface (I)	11.0	11.7	3.3	3.6	12.8	2.9
Fractured surface (F)	26.8	20.3	18.1	17.5	25.9	5.6
F to I ratio	2.44	1.73	5.43	4.84	2.02	1.95

Fig 2 X-ray diffraction analysis of relative intensity (%) of the tetragonal and monoclinic phases on intact (*top*) and fractured (*bottom*) surfaces of a disk specimen of YZ Zirconia (T = tetragonal peak at $2\theta = 30.167$ degrees; M = mm1 (\overline{I} , I, I) at $2\theta = 28.174$ degrees and m2 (I, I, I) at $2\theta = 31.467$ degrees).



tured YZ Zirconia surface contained approximately 5 times as much monoclinic phase compared to that of its intact surface. The fractured IZ and Cercon surfaces contained approximately twice as much monoclinic phase compared to their intact surfaces (Table 3).

SEM images of the fractured surface of IZ revealed platelets of alumina embedded in an amorphous glass matrix. Irregular-shaped conglomerates of approximately 1 to 2 μ m in size appearing to be monoclinic zirconia were also identified (Figs 3 and 4). The etched IZ specimen (Fig 4) provided additional information

about the distribution of zirconia polycrystals, which were not conspicuous in the unetched sample (Fig 3). The crack that transverses the IZ specimen in Fig 4 involved intergranular and transgranular modes of fracture through both alumina and zirconia phases.

For the YZ Zirconia and Cercon specimens, etching did not produce significant differences, and therefore only the SEM images of unetched specimens are shown (Figs 5 and 6). SEM images of the fractured surface of the YZ Zirconia specimen showed a honeycomb appearance representative of closely compacted



Fig 3 SEM image of unetched In-Ceram Zirconia (×4,000) (*solid arrow:* zirconia polycrystal; *dashed arrows:* alumina platelet).



Fig 4 SEM image of etched In-Ceram Zirconia (backscattered image, ×2,000) (*black arrows:* zirconia polycrystal; *arrowheads:* alumina plate; *white arrows* define the pathway of a crack).



Fig 5 SEM image of unetched YZ Zirconia (×7,000) (*black arrows* define the pathway of a crack; *white arrow:* intergranular fracture; *arrowhead:* transgranular fracture).



Fig 7 SEM image of etched Empress 2 specimen (×2,085) (*white arrow:* pinnacle-shaped lithium disilicate crystals; *black arrow:* lithium disilicate crystals embedded in glass matrix).



Fig 6 SEM image of unetched Cercon (×7,000) (*white arrow:* aggregates of tetragonal zirconia granules; *black arrow:* conglomerates appearing to be monoclinic zirconia).

tetragonal zirconia granules of $< 1 \mu m$ in size. The principal mode of failure was intergranular fracture between the zirconia granules, although transgranular fracture was also observed (Fig 5). The fracture surface of Cercon comprised aggregates of tetragonal zirconia granules of $< 1 \mu m$ in size, which were tightly packed. Irregular-shaped conglomerates appearing to be monoclinic zirconia were abundant on the fractured surface (Fig 6).

The etched Empress 2 specimen revealed pinnacleshaped lithium disilicate crystals a few micrometers in length distributed throughout the specimen. The glass matrix was visible where it was not completely etched (Fig 7). The etched Empress 2 specimen showed the lithium disilicate crystals and unetched area with the crystals embedding in the matrix; therefore, the SEM image of the unetched specimens is not shown.

Discussion

Although the ceramic materials tested were core materials, their chemical solubility can still be of concern because clinical situations may require the core ceramic framework to be exposed to the oral environment.⁷ For example, in areas where occlusal clearance is limited, occlusal contacts on the ceramic prosthesis may be designed intentionally to be placed on the core ceramic material. At prosthesis margins, where pulpal vitality and tooth structure conservation are of a higher priority than esthetics, core ceramic material may also be used instead of the veneer porcelain to terminate the margin. The results of this study showed that there were no significant differences in the chemical solubility of the 4 ceramics tested. The chemical solubilities of the 4 ceramics as expressed in mass loss per unit of surface area (µg/cm²) were below the maximum chemical solubility allowance of 2,000 μ g/cm² required of core ceramic material (type I, Class 1 or type II, Class 1) according to the ISO 6872:1995(E) specifications for dental ceramics. The present ISO 6872:1995(E) protocol used 4% acetic acid as the chemical agent to evaluate chemical solubility of the ceramic materials by refluxing the acid for 16 hours. It has been shown in an earlier study that 2 preparations of 99% ZrO, blocks were resistant even to 168 hours of 4% acetic acid reflux without compromising their subsequent flexural strength.¹⁵ The effect of 4% acetic acid used for 1 week at 80°C was likened to immersion in artificial saliva at 22°C for 22 years.¹⁹ Thus, the results of the present study reaffirm the chemical stability of ceramic materials, including the zirconia-based ceramics.

The UFS and BFS of the ceramics tested well exceeded the requirement of 100 MPa for type II, Class 1 ceramics according to ISO 6872:1995 for dental ceramics.¹⁶ Empress 2 was chosen as the control material because of the availability of laboratory studies for comparison, and the documented clinical studies for reference. Empress 2 specimens (lithium disilicate ceramics) had flexural strengths in the 200- to 400-MPa range, which was significantly lower than the zirconiabased ceramics. This observation is generally in line with earlier observations.9,20 The strength of the ceramics is attributed to the densely packed microstructure of lithium disilicate crystals, which was also demonstrated in this study. The concentration of the crystals within the ceramics was reported to be as high as 90%.²¹ The clinical performance of lithium disilicate ceramics as a core material of fixed partial dentures (FPD) had been studied. Two of 30 FPDs fractured within a 2-year observation period, yielding a 93% success rate.²² Although the figure was lower than the published success rate of metal-ceramic FPDs, the investigators were still optimistic about the result. However, the success rates of Empress 2 FPDs were relatively low

in other studies. For instance, a study of 20 anterior and posterior FPDs reported that 50% had "catastrophic failure" during the first 2 years of the study period.²³ Another 5-year study of 31 Empress 2 FPDs also noted that the 50-month survival rate was only 70%.²⁴

The test methods for UFS and BFS are variations of the same theme and produce different results. The different geometry and loading and supporting designs of the specimens produce different fracture patterns. Such tests do not contradict each other, but rather reflect the mechanical properties of these materials under different conditions. All 3 zirconia-based ceramics showed significantly higher flexural strength than the lithium disilicate ceramics. In particular, YZ Zirconia, an yttria-stabilized zirconia ceramic, showed the highest UFS (899 MPa) and BFS (1,107 MPa) among the zirconia-based ceramics. Such strength is comparable to those developed by experimental yttriastabilized zirconia ceramics prepared by dry pressing and pressureless sintering in air¹² and an isostatically hot-pressed sintered 5% yttria-stabilized zirconia ceramic prepared with a CAD/CAM system.³

Similar to YZ Zirconia, Cercon is an yttria-stabilized zirconia ceramic and is also fabricated by sintering of CAM-milled presintered zirconia blanks. However, its mean flexural strengths (UFS: 458 MPa; BFS: 927 MPa) were significantly lower than those of YZ Zirconia in the present study. The UFS (409 MPa) of IZ was comparable to that of Cercon, but its BFS (523 MPa) was significantly lower than that of Cercon. Factors that may have affected the strength of yttria-stabilized zirconia ceramics include the density of the presintered pressed powder blocks as it relates to critical flaw size population, the sinterability of the pressed powder as it relates to the initial particle size, the yttria content as it relates to the amount of tetragonal to monoclinic phase transformation, and mechanically induced flaws and residual compressive stresses during specimen preparation.^{12,25} The extent to which each of these factors contributed to the differences in strength between YZ Zirconia and Cercon remains to be studied. However, it was evident from this study that the tetragonal to monoclinic phase transformation, which was considered the major toughening mechanism, occurred at the fracture interface of all 3 zirconia-based ceramics (Table 3). It was observed that the relative amount of monoclinic phase present on the intact surface of Cercon bar specimens (12.8%) was significantly higher than that on intact Cercon disk specimens (2.9%). In contrast, the relative amounts of monoclinic phase present on the bar and disk specimens of IZ and YZ Zirconia were similar. Further study is necessary to understand why intact Cercon bar specimens had such a high relative amount of monoclinic phase. Studying the ratio of the relative amount of monoclinic phase

present at the fractured surface compared to that on the intact surface (F to I ratio) may help explain the variation in strength among the 3 zirconia-based ceramics. The F to I ratio of YZ Zirconia was approximately 5, while that of IZ and Cercon was approximately 2. Thus, tetragonal to monoclinic phase transformation happened more readily on the fractured surface of YZ Zirconia, which may explain its superior strength.

The availability of tetragonal phase in the zirconiabased ceramics, in turn, can also be affected by how the specimens were prepared. Air abrading the surface of stabilized zirconia ceramics with 110-µm alumina particles was found to increase the relative content of monoclinic phase in comparison with surface treatment by dry or wet grinding with 50- and 150-µm diamond burs.¹² Excessive heat produced during machining may cause local temperature to exceed 700°C when the reverse monoclinic to tetragonal phase transformation occurs.²⁶ Low-temperature aqueous aging of yttria-stabilized zirconia ceramic is known to promote tetragonal to monoclinic phase transformation in addition to causing extensive microcracking.²⁷ The present study limited surface treatment of the ceramics to finishing with 600-grit silicon carbide abrasive paper, and hard machining on the specimens was not necessary. All specimens were prepared and tested in a dry condition. Thus, the influence that such factors had on the result of the present study is negligible.

Although the present study showed that such zirconiabased materials are promising, it is unknown how the addition of veneer porcelain can change their physical properties.²⁸ Further studies on these zirconia-based materials laminated with veneer porcelain could be carried out.

Summary

The chemical solubility, UFS, and BFS of 3 zirconiabased ceramics were tested against a control lithiumdisilicate ceramic. The chemical solubilities of the ceramics tested were not significantly different. They all satisfied the chemical solubility allowance required of core ceramic material (type I, Class 1 or type II, Class 1) according to ISO 6872:1995(E) specifications on dental ceramic. UFS and BFS of the lithium disilicate ceramics were significantly lower than those of the 3 zirconia-based ceramics. An yttria-stabilized zirconia ceramic (YZ Zirconia) possessed significantly higher UFS and BFS than another yttria-stabilized zirconia ceramic (Cercon) and a zirconia-reinforced, glass-infiltrated alumina ceramic (IZ). The UFS values of Cercon and IZ were not significantly different from each other, while the BFS of Cercon was significantly higher than that of IZ. Based on the findings of the present study, these high-strength zirconia-based ceramics could have promising clinical applications.

References

- Swain MV. Toughening mechanisms for ceramics. Mater Forum 1989;13:237–253.
- Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part I. Pressable and alumina glass-infiltrated ceramics. Dent Mater 2004;20:441–448.
- Guazzato M, Albakry M, Ringer SP, Swain MV. Strength, fracture toughness and microstructure of a selection of all-ceramic materials. Part II. Zirconia-based dental ceramics. Dent Mater 2004;20:449–456.
- Chong KH, Chai J, Takahashi Y, Wozniak W. Flexural strength of In-Ceram alumina and In-Ceram zirconia core materials. Int J Prosthodont 2002;15:183–188.
- Chong KH, Chai J. Probability of failure of veneered glass-fiberreinforced composites and glass-infiltrated alumina with or without zirconia reinforcement. Int J Prosthodont 2003;16:487–492.
- Esquivel JF, Chai J, Wozniak WT. The physical properties of lowfusing porcelains for titanium. Int J Prosthodont 1996;9:563–571.
- Esquivel-Upshaw JF, Chai J, Sansano S, Shonberg D. Resistance to staining, flexural strength, and chemical solubility of core porcelains for all-ceramic crowns. Int J Prosthodont 2001;14:284–288.
- Apholt W, Bindl A, Lüthy H, Mörmann. Flexural strength of Cerec 2 machined and jointed In-Ceram-Alumina and In-Ceram-Zirconia bars. Dent Mater 2001;17:260–267.
- Tinschert J, Natt G, Mautsch W, Augthun M, Spiekermann H. Fracture resistance of lithium disilicate-, alumina- and zirconiabased three-unit fixed partial dentures: A laboratory study. Int J Prosthodont 2001;14:231–238.
- Guazzato M, Albakry M, Swain MV, Ironside J. Mechanical properties of In-Ceram Alumina and In-Ceram Zirconia. Int J Prosthodont 2002;15:339–346.
- 11. Lothar L. Cercon–The all-ceramic CAM system by Degussa. Quintessence Int 2001;52:811–814.
- Kosmač T, Oblak C, Jevnikar P, Funduk N, Marion L. Strength and reliability of surface treated Y-TZP dental ceramics. J Biomed Mater Res 2000;53:304–313.
- Luthardt RG, Holzhüter M, Sandkuhl O, et al. Reliability and properties of ground Y-TZP-zirconia ceramics. J Dent Res 2002;81:487-491.
- Li J, Liao H, Hermansson L. Sintering of partially-stabilized zirconia and partially-stabilized zirconia-hydroxyapatite composites by hot isostatic pressing and pressureless sintering. Biomaterials 1996;17:1787–1790.
- Ardlin BI. Transformation-toughened zirconia for dental inlays, crowns and bridges: Chemical stability and effect of low-temperature aging on flexural strength and surface structure. Dent Mater 2002;18:590–595.
- International Organization for Standardization. ISO/DIS 6872: Dental ceramic. Geneva: ISO, 1995.
- International Organization for Standardization. ISO 3696:1987: Water for analytical laboratory use–Specification and test methods. ISO: Geneva, 1987.
- Garvie RC, Nicholson PS. Phase analysis in zirconia systems. J Am Ceram Soc 1972;55:303–305.
- DeRijk WG, Jennings KA, Menis DL. A comparison of chemical durability of test solutions for dental porcelains. In: Sauer BW (ed). Biomedical Engineering–20. Recent Developments. Proceedings, Southern Biomedical Engineering Conference. New York: Pergamon, 1985:152–155.
- Albakry M, Guazzato M, Swain MV. Biaxial flexural strength and microsructure changes of two recycled pressable glass ceramics. J Prosthodont 2004;13:141–149.
- Cattell MJ, Palumbo RP, Knowles JC, Clarke RL, Samarawickrama DYD. The effect of veneering and heat treatment on the flexural strength of Empress 2 ceramics. J Dent 2002;30:161–169.

- Esquivel-Upshaw JF, Anusavice KJ, Young H, Jones J, Gibbs C. Clinical performance of a lithia disilicate-based core ceramic for three-unit posterior FPDs. Int J Prosthodont 2004;17:469–475.
- Taskonak B, Sertgoz A. Two-year clinical evaluation of lithia-disilicate-based all-ceramic crowns and fixed partial dentures. Dent Mater 2006;22:1008–1013.
- Marguardt P, Strub JR. Survival rates of IPS Empress 2 all-ceramic crowns and fixed partial dentures: Results of a 5-year prospective clinical study. Quintessence Int 2006;37:253–259.
- Gross V, Swain MV. Mechanical properties and microstructure of sintered and hot isotatically pressed yttria-partially-stabilised zirconia (Y-PSZ). J Aust Ceram Soc 1986;22;1–12.
- Swain MV, Hannink RHJ. Metastability of the martensitic transformation in a 12 mol% ceria-zirconia. J Am Ceram Soc 1989;72:1358-1364.
- Kim DJ. Influence of aging environment on low-temperature degradation of tetragonal zirconia alloys. J Eur Ceram Soc 1997;17:897–903.
- Chu FC, Frankel N, Smales RJ. Surface roughness and flexural strength of self-glazed, polished and reglazed In-Ceram/Vitadur alpha porcelain laminates. Int J Prosthodont 2000;13:67–71.

Literature Abstract

Treatment history of teeth in relation to the longevity of the teeth and their restorations: Outcomes of teeth treated and maintained for 15 years

This study evaluated a tooth's treatment history and the longevity of its restoration and compared and contrasted the survival of teeth with and without restorations, including the survival of teeth with extensive restorations. Data were collected for 3,071 teeth from 148 fully compliant patients from 1 private practice. Follow-up times ranged from 15 to 23 years (mean: 19.2 years). Patients had to meet defined criteria to be enrolled in the study. Treatments were categorized as follows: unrestored, surface restoration (1, 2, 3, or 4+), complete crown, abutment for FPD, abutment for RPD, and root canal treatment. Failure modes were as follows: restorative failure, extraction, and any failure (restorative failure or extraction). Caries risk assessment was also performed for all patients. Multivariate survival analysis was used for data analysis ($\alpha = .05$). The results showed that unrestored teeth had the best overall survival when compared with restored teeth. Teeth with 3 to 5 surface restorations were 4 times more likely to fail than unrestored teeth. Complete crowns and abutments for FPDs had fewer restorative failures compared to teeth with complex multisurface restorations. RPD abutments experienced the highest failure rate compared with restored teeth. It was demonstrated that failed teeth had a greater *S mutans* level, greater *Lactobacillus* level, higher dietary frequency per day, and lower salivary buffer capacity. The results support the need for full-crown coverage to improve the prognosis of teeth restored with multisurface restorations.

Miyamoto T, Morgano SM, Kumagai T, Jones JA, Nunn ME. *J Prosthet Dent* 2007;97:150–156. References: 25. Reprints: Dr Martha E. Nunn, Boston University School of Dental Medicine, Department of Health Policy and Health Service Research, 650 Harrison Ave, Boston, MA 02118. Fax: 617 414 1061—*Majd Al Mardini, Hamilton, Canada* Copyright of International Journal of Prosthodontics is the property of Quintessence Publishing Company Inc. and its content may not be copied or emailed to multiple sites or posted to a listserv without the copyright holder's express written permission. However, users may print, download, or email articles for individual use.