Published in final edited form as:

Dent Mater. 2013 December ; 29(12): 1201–1208. doi:10.1016/j.dental.2013.09.004.

Edge chipping and flexural resistance of monolithic ceramics[☆]

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Abstract

Objective—Test the hypothesis that monolithic ceramics can be developed with combined esthetics and superior fracture resistance to circumvent processing and performance drawbacks of traditional all-ceramic crowns and fixed-dental-prostheses consisting of a hard and strong core with an esthetic porcelain veneer. Specifically, to demonstrate that monolithic prostheses can be produced with a much reduced susceptibility to fracture.

Methods—Protocols were applied for quantifying resistance to chipping as well as resistance to flexural failure in two classes of dental ceramic, microstructurally-modified zirconias and lithium disilicate glass–ceramics. A sharp indenter was used to induce chips near the edges of flat-layer specimens, and the results compared with predictions from a critical load equation. The critical loads required to produce cementation surface failure in monolithic specimens bonded to dentin were computed from established flexural strength relations and the predictions validated with experimental data.

Results—Monolithic zirconias have superior chipping and flexural fracture resistance relative to their veneered counterparts. While they have superior esthetics, glass–ceramics exhibit lower strength but higher chip fracture resistance relative to porcelain-veneered zirconias.

Significance—The study suggests a promising future for new and improved monolithic ceramic restorations, with combined durability and acceptable esthetics.

Keywords

Edge chipping; Flexural fracture; Monolithic restorations; Zirconia-based ceramic; Glass–ceramic; Graded glass–zirconia

[☆]Certain commercial materials are identified in this paper to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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Conflict of interest

All authors declare no conflict of interest.

1. Introduction

More than a decade of clinical trials recording survival rates for all-ceramic posterior crowns and fixed dental prostheses (FDPs) indicate vulnerabilities to various failure modes [1–24]. The trend in circumventing this problem has been toward a strong and tough yttria tetragonal zirconia polycrystal (Y-TZP) core veneered with an esthetic porcelain [25–27]. Such bilayer systems have several major drawbacks: (i) their fabrication is a multistep process; (ii) the veneer has a low toughness and is consequently susceptible to chipping [28]; (iii) the bonding between veneer and core can be weak relative to the toughness of the constituent material layers, with ensuing potential for delamination [29,30] and (iv) residual tensile stresses can develop during the veneering process, further degrading the porcelain and its bond to the zirconia core [31–34]. Veneered zirconia prostheses do not appear to perform as well as their veneered metal counterparts [9,27,28].

The obvious way to circumvent all these drawbacks is to replace the veneer/core bilayer with a monolithic ceramic. This has not been straightforward, because the microstructural qualities that confer good mechanical properties do not lend themselves to good esthetics, and vice versa. However, some approaches are emerging with the promise of improved clinical performance. One such is to begin with translucent but inherently weak glass–ceramics, such as Dicor [35,36], and to refine the compositions and microstructures to produce a tougher ceramic without compromising the esthetics. The lithium disilicate IPS e.max glass–ceramics manufactured by Ivoclar-Vivadent fall into this category [37]. Those materials have strong needle-like crystals embedded within a glass matrix, mimicking natural enamel and thereby inhibiting crack propagation. They have performed well in crown applications [13,23]. However, they are not as strong as the zirconias, and are less suitable for applications where stress concentrations can be high, e.g. FDP connectors [38].

An alternative approach has been to begin with a strong and dense zirconia, and to manipulate additive components and heat treatments to produce an acceptable translucency. The monolithic zirconias BruxZir by Glidewell and LAVA Plus by 3 M ESPE are examples. The translucency of BruxZir is achieved via the elimination of light-scattering alumina sintering aids and porosities, along with the utilization of a higher sintering temperature (1530 °C) and longer dwell time (6 h). The translucency of LAVA Plus is attained by reducing alumina sintering aids, but also by increasing the density of the green compact in order to reduce the sintering temperature (1450 °C) and dwell time (2 h), with resultant finer grain size. Glass-infiltrated zirconia (GZG) is another route to improved translucency [39]. While remaining highly fracture resistant, zirconia-based ceramics do not yet match the esthetics of the glass–ceramics.

In assessing potential lifetimes of any material type it is important to consider the different modes in which fracture may occur. Foremost among these modes is top-surface chipping [40–42] and subsurface flexural fracture [43–45]. Resistance to one mode does not necessarily imply resistance to the other. The hypothesis in this work was that monolithic crowns can sustain uncommonly high bite forces, provided certain dimensional requirements are met. To test that hypothesis, the chipping resistance and flexural strength were evaluated for candidate lithium disilicate glass–ceramics and modified zirconia materials. To place the

results into perspective, comparative data for a veneering porcelain and unmodified Y-TZP were also evaluated.

2. Materials and methods

2.1. Material selection and specimen preparation

The restorative materials selected for this study are listed in Table 1, along with values of Young's modulus E , toughness T and strength S . The properties data for commercial veneering porcelain, as well as for Y-TZP and glass-infiltrated zirconia (GZG), have been documented in earlier reports [42,46,47].

Zirconia plates were fabricated in-house from 3 mol% Y-TZP powder (TZ-3Y-E grade, Tosoh, Tokyo, Japan) and sintered at 1450 °C for 2 h in air at a heating/cooling rate of 10 °C/min. Glass-infiltrated zirconia (GZG) plates were produced by presintering the same Y-TZP powder compacts at 1350 °C for 1 h followed by infiltration at 1450 °C for 2 h to a depth of 100 µm with a glass of matching coefficient of thermal expansion, at a heating/cooling rate of 14 °C/min [39,48]. All specimens were fabricated as plates of lateral dimension 10 mm × 10 mm. Those to be used for chipping tests were 2 mm thick ($n = 4$, each material). Those for flexural testing were prepared with thicknesses 1 mm, 1.5 mm and 2 mm ($n = 6$, each material and each thickness). One set was prepared with thickness 0.5 mm and subsequently veneered with porcelain (VM9, Vita, Bad Säckingen, Germany) by Marotta Dental Laboratories to provide porcelain/zirconia bilayers of total thicknesses 1.0 mm, 1.5 mm and 2.0 mm ($n = 6$, each thickness).

Lithium disilicate glass-ceramics were obtained from the manufacturer (IPS e.max, Ivoclar-Vivadent, Amherst, NY). These came in two forms, Press and CAD, reflecting differences in processing condition [37]. The IPS e.max Press ingots were heat-pressed at 915 °C for 15 min in a EP 600 furnace (Ivoclar Vivadent) and then divested. A reaction layer from the heat treatment was removed by immersing pressed specimens in an aqueous solution containing 0.6% hydrofluoric acid and 1.7% sulphuric acid followed by blasting with Al₂O₃ particles (100 µm at 0.2 MPa pressure). The IPS e.max CAD ingots were first heat treated to form partially crystallized glass-ceramic blocks, and then were CAD/CAM machined to shape. These were then heated to 840 °C for 7 min for final crystallization. Specimens were fabricated as bars 2 mm × 3 mm × 25 mm for chipping ($n = 6$, each material) and flexure ($n = 20$, each material) testing. Additional IPS e.max CAD plate specimens 10 mm × 10 mm were prepared for flexure testing ($n = 20$).

Data for a common dental cement (Multilink Automix, Dual-Cure, Ivoclar-Vivadent) used to bond zirconia-based and lithium disilicate specimens to a 4 mm thick composite base (Z100, 3 M ESPE, St. Paul, MN), as well as for natural dentin itself, are included in Table 1. This composite base was used to stabilize the specimens in the chipping tests, and to provide a dentin-like support substrate in the flexure tests [49,50]. Prior to cementation, top and adjacent side surfaces of the ceramic plates for the chipping tests were first polished to 1 µm finish. The intaglio surfaces of the zirconia plates for flexure tests were likewise pre-polished, while those for the lithium disilicate and GZG plates were pre-etched (4% HF for 30 s and 5 min, respectively).

Scanning electron microscope images of each restorative material were then taken to reveal the microstructures. Sections of the specimens selected for examination were polished to 1 μm , and then either thermally etched (zirconia, 1250 $^{\circ}\text{C}$ for 20 min) or chemically etched (4% hydrofluoric acid –20 s for e.max Press and CAD, 30 s for porcelain, 5 min for GZG).

2.2. Critical forces to produce chipping

Following previous protocols used to evaluate the Y-TZP, GZG and porcelain materials listed in Table 1, chipping tests were carried out on the lithium disilicate bars [42]. A Vickers diamond pyramid was located normally at specified distances h from a side wall, at crosshead speed 0.1 mm min^{-1} , with indentation diagonals parallel and perpendicular to the edge. The critical forces P_C at which a chip spalled off the side wall were then recorded for each indent. These forces are directly proportional to the ceramic toughness T , as expressed in the chipping relation [51].

$$P_C = \beta T h^{3/2} \quad (1)$$

where $\beta = 9.3$ is a dimensionless constant.

2.3. Critical forces to produce flexural fracture

Estimates of the capacity for each material system to sustain flexural stress from the applied loading were calculated using documented flexure formulae for flat-layer systems cemented onto dentin-like substrates. Flexure tests were performed on a universal testing machine (Model 5566, Instron, Norwood, MA), with contact load applied at the top surface with a hard tungsten carbide spherical indenter at crosshead speed 1 mm min^{-1} [52,53]. A thin plastic sheet was placed between the indenter and the ceramic surface to prevent fracture from spurious contact-induced top-surface cone cracks. The critical load for the onset of radial cracking at the intaglio surface was recorded as a load drop. This kind of subsurface fracture occurs when the tensile stress at the intaglio surface of the ceramic layer equals the strength S of the material. (The radial cracks continue to expand outward at the inner surface of the ceramic layer with further increase in load, ultimately breaking through to the top surface [54].) For a monolithic ceramic layer (C) of thickness d and modulus E_C on a compliant substrate (S) of modulus E_S , the critical loads for flexural fracture are given by [53].

$$P_F = \frac{1.35 S d^2}{\log(E_C/E_S)} \quad (2)$$

Likewise, for a veneer/ceramic (V/C) bilayer of net thickness $d = d_V + d_C$ free of any residual expansion mismatch stresses on the same compliant substrate (S), the corresponding critical loads are [53].

$$P_F = \frac{1.35 S d^2}{(E_C/E^*) \log(E^*/E_S)} \quad (3)$$

The quantity E^* is a composite veneer/ceramic modulus given by $E^* = E_C \{ 1 + \varepsilon^2 \delta^3 + \varepsilon \delta (5.66 + 2.18 \delta) \} / \{ 1 + 1.97 \delta + \varepsilon \delta [3.69 + 2.18 \delta + \delta^2] \}$, with modulus ratio $\varepsilon = E_V/E_C$ and thickness ratio $\delta = d_V/d_C$. Eqs. (2) and (3) have been validated by extensive experimentation and finite element analysis [53].

Evaluation of the critical loads P_F in Eqs. (2) and (3) requires the presence of the cementation interlayer to be taken into account. One may consider the interlayer (I) of thickness t to be an integral part of the underlying dentin substrate (D), with a composite substrate modulus $E_S = E_I(E_D/E_I)^L$, with $L = \exp\{-[1.18 + 0.33 \log(t/d)]^3\}$ a dimensionless constant [55].

Accordingly, using material properties from Table 1, critical loads for flexure-induced fracture were calculated from Eq. (2) for monolithic ceramic layers of thickness $d = 1$ mm, 1.5 mm and 2 mm and cementation thickness $h = 50$ μm . In the case of porcelain-veneered zirconia, Eq. (3) was used, again with net thickness $d = 1$ mm, 1.5 mm and 2 mm and cementation thickness $h = 50$ μm but with fixed core thickness $d_C = 0.5$ mm.

3. Results

Scanning electron micrographs are shown in Fig. 1 for Y-TZP, GZG, lithium disilicate and porcelain. In contrast to the homogeneous, equiaxed fine-grain structures of the zirconia-based ceramics, lithium disilicate Press and CAD and porcelain have a glass matrix containing $\approx 70\%$ needle-like elongate and $\approx 30\%$ equiaxed coarse crystallites, respectively. In the Press grade the crystallites are ≈ 4 μm long and ≈ 0.6 μm wide and somewhat aligned parallel to the polished surface, whereas in the CAD grade the crystallites are ≈ 1 μm long and ≈ 0.4 μm wide and more randomly oriented. The Press grade exhibits slightly higher toughness, because of the greater impedance to crack propagation by the larger grains, but slightly lower strength, because these same grains introduce larger starting flaws into the structure (Table 1).

The results for the chipping tests adjacent to the orthogonal faces are shown as plots of critical load P_C versus indent location h for each monolithic ceramic in Fig. 2. The data for Y-TZP, GZG and porcelain are taken from previous studies [42]. Student's t -tests comparing individual values of $P/h^{3/2}$ evaluated from the data points in Fig. 2 indicate no statistically significant difference between Y-TZP and GZG or between Press and CAD ($p < 0.05$); but significant differences between zirconia-based ceramics and glass-ceramics, and glass-ceramics and porcelain ($p < 0.00001$). The quantities T listed in Table 1 are 'chipping toughnesses' evaluated by inserting mean and standard deviation $P/h^{3/2}$ values for each material into Eq. (1). The solid lines are representations of Eq. (1) using averaged toughnesses $T = 3.70$ MPa $\text{m}^{1/2}$ for Y-TZP and GZG zirconia-based ceramics, $T = 1.40$ MPa $\text{m}^{1/2}$ for Press and $T = 1.31$ MPa $\text{m}^{1/2}$ for CAD glass-ceramics, and $T = 0.94$ MPa $\text{m}^{1/2}$ for porcelain, from Table 1. The horizontal dashed line in Fig. 2 represents a nominal maximum biting force of 400 N for human molars [43].

Fig. 3 compares results for calculated and experimentally determined critical flexure loads P_F for each ceramic system cemented to its dental composite base, at three values of net ceramic thickness d . The theoretical values were estimated using values of strength S and

modulus E in Table 1 with a cement layer thickness of 50 μm . The uncertainty bars are one standard error ($n = 6$). The horizontal dashed line represents a nominal maximum biting force of 400 N for human molars. At any given thickness, the lithium disilicate glass–ceramics have a lower critical load for radial fracture relative to their zirconia-based counterparts, either monolithic or porcelain-veneered. The advantage of maintaining a thick ceramic crown layer is apparent, with a quadratic (d^2) dependence in Eqs. (2) and (3). For any given material system, the difference between theoretical prediction and experimental data sets for each material system was insignificant, as verified by a nonparametric one-sample Kolmogorov–Smirnov test comparing means and standard errors ($p < 0.05$). An exception was the data for the 2 mm thick GZG plates; in this case, re-examination of the broken specimens revealed premature failure from top-surface cone cracking, explaining the discrepancy and attesting to the ultra-high strength of GZG [56].

4. Discussion

This study has evaluated the mechanical properties of the latest-generation monolithic ceramics for crowns and bridges, with specific attention to zirconia-based ceramics and lithium disilicate glass–ceramics. The zirconia-based materials have markedly superior resistance to chipping and flexural fracture. However, even the most translucent zirconias produced to date cannot approach the esthetics of glass–ceramics, especially in terms of shade matching. The traditional procedure of adding a porcelain veneer to a zirconia core overcomes this problem, but brings with it all the drawbacks alluded to in the Introduction, including enhanced susceptibility to chipping. (An independent in vitro edge-chipping study on porcelain-veneered zirconias using sharp conical indenters reports an edge-chipping resistance similar to that of the monolithic porcelain shown in Fig. 2 [41].) Glass–ceramics, on the other hand, are less vulnerable to chipping, but cannot match the zirconias for fracture resistance. A major advantage of glass–ceramics is that they can be readily etched and silanized at the intaglio surfaces, with attendant superior bonding to a support substrate. Glass–ceramics also exhibit similar wear behavior to enamel [57]. It is the quest for ceramics with combined superior mechanical properties and acceptable esthetics that has driven materials scientists to develop translucent zirconias, either by refining the microstructure or by infusing with glass [39], without compromising strength and toughness [42,47].

The work presented in this paper presents a simple route to quantifying the vulnerabilities of restorative ceramic systems to two major sources of failure, occlusal-surface chipping and cementation-surface flexural fracture. The evaluations pertain to idealized, flat-layer specimen geometries, employed to enable the application of explicit fracture relations in terms of characteristic prosthesis dimensions and material properties. It is of interest to note that while the critical load for chipping in Eq. (1) is governed by toughness T , the fracture loads in flexure in Eqs. (2) and (3) are governed by strength S . This is because chipping involves a stage of stable crack propagation prior to final spallation [51], while flexural fracture involves abrupt radial crack initiation at a critical tensile stress [50]. Since T and S do not always scale in the same way, close attention needs to be paid to both properties in assessing performance. In the case of the glass–ceramics, for instance, an elongate grain structure is well suited to providing a respectable toughness, by inhibiting crack

propagation, but at the same time limits the strength, by promoting crack initiation within a relatively coarse microstructure (see Table 1) [36].

Of the two fracture modes, it is chipping that is most likely to occur at lower occlusal loads. Note that all the experimental critical loads in Fig. 2 lie below the dashed line representing a nominal maximum biting force of 400 N. The data in that figure suggest that dental prostheses should be fabricated to avoid sharp contacts closer than about 1 mm from the side walls. On the other hand, the critical loads for flexural fracture in Fig. 3 all lie above the same representative biting force. This suggests that most crown systems will be immune to flexural fracture from occlusal overloading, provided the ceramic thickness lies above 1 mm. The data in Figs. 2 and 3 also provide a graphic means for ranking the fracture resistance of prospective monolithic prosthetic ceramics. In all cases, the critical chipping and flexural fracture loads P_C and P_F for lithium disilicate fall below those for their Y-TZP and GZG counterparts, confirming that the zirconia-based materials remain the most robust monolith ceramics. However, the value of P_C for lithium disilicate in Fig. 2 is higher than that of porcelain, indicating a superior resistance to chipping relative to veneered core materials.

It is acknowledged that the flat-layer configurations investigated here are an oversimplification of dental prosthesis geometries. In fact, the critical loads P_C for chipping at large distances from the side walls are somewhat lower on the curved surfaces of anatomically-correct ceramic crowns than on their flat counterparts, i.e. experimental data would fall below the predicted linear fits at large h in Fig. 2 [42]. For radial fracture in curved surfaces, increased curvature actually increases the critical loads P_F for initiation of flexural fracture, as the crown takes on a dome-like configuration [54]. Increased curvature also makes it easier for popped-in radial cracks to propagate around the side walls to the crown margins with further increased occlusal load, meaning that smaller crowns are more likely to fail after radial crack initiation [58,59]. Regardless, the flexure testing of flat ceramic plates bonded onto a compliant substrate is much closer to clinical configurations than conventional three-point bend tests on isolated bar specimens. Notwithstanding such geometrical complications, data shifts due to specimen curvature are likely to be common to all dental ceramics, suggesting that the relative positions of the fitted line in Fig. 2 and heights of the bars in Fig. 3 remain an accurate comparative measure of fracture vulnerability in crown structures. The situation becomes more complex in the case of FDPs where stress concentrations may occur, especially where the prosthesis becomes thinner, e.g. at connector sites. FDPs with frameworks made of relatively low strength lithium disilicate glass–ceramics require larger connector areas to meet load-bearing requirements relative to their stronger zirconia counterparts [20,37]. This differs from single crowns, where the prosthesis is firmly supported by underlying dentin.

Finally, we would point out that the current experimental P_C and P_F data in Figs. 2 and 3 were obtained using relatively fast loading rates, fast enough to avoid significant effects of so-called ‘slow crack growth’ from chemical interactions between cracks and intrusive moisture [60]. They were also conducted in single-cycle loading, so that mechanical fatigue was not a factor [61–63]. In reality, critical fracture loads can be diminished by a factor of two or more over extended chewing cycles [64–66]. Nevertheless, the test procedures

described here provide useful guidelines to ranking performance of different dental material systems.

5. Conclusions

Monolithic ceramic restorations exhibit markedly superior fracture resistance relative to their porcelain-veneered counterparts, from both chipping and radial cracking modes. Zirconia-based ceramic monoliths have higher resistance to failure than lithium disilicate glass-ceramics, but are less esthetic. However, recent developments in sintering treatments and infiltration procedures are producing more translucent zirconia systems, signaling a bright future for monolithic zirconia-based prostheses. Simple chipping and flexure test procedures usefully quantify the resistance to potential fracture for crown and PDF structures, and are amenable to prediction from explicit fracture relations.

Acknowledgments

The authors would like to thank Dr. F.K. Fatehi for performing statistical analysis. Funding was provided by the United States National Institute of Dental and Craniofacial Research (Grant 2R01 DE017925) and the National Science Foundation (Grant CMMI-0758530) and by NIST.

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Fig. 1.

Scanning electron micrographs, showing microstructures of candidate materials: thermally etched (a) Y-TZP, and hydrofluoric acid (4%) etched (b) graded glass/zirconia/glass (GZG), (c) lithium disilicate glass-ceramic (Press), (d) lithium disilicate glass-ceramic (CAD) and (e) feldspathic porcelain (VM9). Acid etching removes the glass phase in graded glass/zirconia/glass and lithium-disilicate glass-ceramics, and also etches away the leucite crystals at a faster rate than the glass matrix in porcelain.

Surfaces gold coated.

Fig. 2.

Critical loads P_C to produce chips as function of distance h from specimen edge, for flat specimens with orthogonal side walls (inset). Data for GZG ($n = 17$) and Y-TZP ($n = 9$) and monolithic porcelain ($n = 11$) are from previous study [42]. Data for lithium disilicate glass–ceramics are from present experiments. Solid lines are predictions from Eqs. (2) and (3). The horizontal dashed line represents a nominal maximum biting force of 400 N for human molars.

Fig. 3.

Critical loads P_F to cause radial fracture at inner surface from flexural bending of ceramic layer on cement/dentin substrate. Theoretical predictions (filled bars) are from Eqs. (2) and (3), using material data from Table 1, for common cementation thickness $50\ \mu\text{m}$. Experimental measurements (unfilled bars) are from tests on ceramic plates cemented to dental composite substrates. Shown for net ceramic thicknesses d indicated, and for fixed zirconia core thickness $d_C = 0.5\ \text{mm}$ in the case of porcelain-veneered zirconia. Uncertainty bars indicate standard errors ($n = 6$). The horizontal dashed line represents a nominal maximum biting force of $400\ \text{N}$ for human molars.

Table 1

Properties of dental materials, mean (standard deviation), *n* tests.

Material	Manufacturer	Modulus <i>E</i> (GPa)	Toughness <i>T</i> (MPa·m ^{1/2})	Strength <i>S</i> (MPa)
Zirconia (3 mol% Y-TZP)	In-house	216 (4) ^a	3.65 (0.2) ^b	1050 (113) ^c
GZG (graded glass/zirconia/glass)	In-house	210 (4) ^a	3.76 (0.3) ^b	1370 (84) ^c
Lithium disilicate (Press)	Ivoclar-Vivadent	95 (5) ^f	1.40 (0.13) ^b	281 (20) ^d
Lithium disilicate (CAD)	Ivoclar-Vivadent	95 (5) ^f	1.31 (0.17) ^b	376 (57) ^d
Feldspathic porcelain (Vita VM9)	Vita Zahnfabrik	66 (3) ^g	0.94 (0.13) ^b	100 (12) ^g
Dental cement (Multilink Automix)	Ivoclar-Vivadent	7.0 (0.4) ^f	–	–
Composite (Z100)	3 M	16 (1) ^h	1.4 (0.1) ^h	120 (12) ^h
Human dentin		18 (2) ^e	–	–

^aIn-house measured using ultrasonic method (*n* = 6 per material).

^bIn-house measured using edge chipping method (minimum *n* = 9 tests per material).

^cIn-house measured using 3-point bend test (*n* = 10 per material).

^dIn-house measured using 4-point bend test (*n* = 20 per material).

^eIn-house measured using nanoindentation method (*n* = 50 indentations).

^fMaterial data sheet from Ivoclar Vivadent.

^gMaterial data sheet from Vita Zahnfabrik.

^hMaterial data sheet from 3 M.