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Interfacial toughness of bilayer dental ceramics based on a short-bar, chevron-notch test

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Abstract

Objective—The objective of this study was to test the null hypothesis that the interfacial toughness of each of two types of bonded core-veneer bilayer ceramics is not significantly different from the apparent fracture toughness of the control monolithic glass veneer.

Methods—T-shaped short bars of a lithia-disilicate glass-ceramic core (LC) and yttria-stabilized polycrystalline zirconia core ceramic (ZC) were prepared according to the manufacturer's recommendations. V-shaped notches were prepared by using 25- μ m-thick palladium foil, leaving the chevron notch area exposed, and the bars were veneered with a thermally compatible glass veneer (LC/GV and ZC/GV). Additionally, we also bonded the glass veneer to itself as a control group (GV/GV). Specimens were kept in distilled water for 30 days before testing in tension. Eight glass veneer bars were prepared for the analysis of fracture toughness test using the indentation-strength technique.

Results—The mean interfacial toughness of the LC/GV group was 0.69 [0.11] MPa·m^{1/2}, and did not significantly differ from that of the GV/GV control group, 0.74 (0.17) MPa·m^{1/2} ($p > 0.05$). However, the difference between the mean interfacial toughness of the ZC/GV group, 0.13 (0.07) MPa·m^{1/2}, and the LC/GV and the GV/GV groups was statistically significant ($p < 0.05$).

Significance—For bilayer all-ceramic restorations with high-strength core materials, the veneering ceramics are the weakest link in the design of the structure. Since all-ceramic restorations often fail from chipping of veneer layers or crack initiation at the interface, the protective effects of thermal mismatch stresses oral prosthesis design should be investigated.

Keywords

chevron notch; bilayer; interfacial toughness; zirconia; all-ceramic

Introduction

All-ceramic restorations are desired by patients and dentists because of their esthetics and biocompatibility. Recently, new dental ceramic core systems with high strength and high toughness were introduced as alternatives to metals. However, chipping and fracture of veneer

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layers have been reported as the failure of some all-ceramic systems [1–11], which raises uncertainty on the benefits of using these all-ceramic prostheses.

Glass-ceramics are polycrystalline materials, whose crystallization by nucleation and crystal growth are controlled by special heat treatment [12,13]. Because of the small amount of glass phase, these materials tend to be relatively pore-free. Recently, two glass-ceramics containing 70 vol% of elongated lithia disilicate crystals have been introduced as core ceramics (Empress® 2 and e.max® Press, Ivoclar-Vivadent, Schaan, Liechtenstein). Ceramic restorations that are made from glass-ceramics are more translucent than purely crystalline ceramics because of their glassy phases. However, because of their low fracture toughness, these systems are limited to three-unit fixed partial dentures that replace a missing tooth anterior to the second premolar [14].

Polycrystalline dental ceramics such as alumina and zirconia have no glassy phase, and they are relatively opaque compared with ceramics that contain a glass phase. Therefore, these stronger and tougher materials are used as substructures (cores) to support weaker but more translucent glass veneers. The ceramics in these systems include a densely sintered high-purity alumina and partially stabilized zirconia that can be milled to dense anatomic shapes by a CAD-CAM device in either partially sintered or fully dense form [15].

A shear test is frequently used to measure the bond strength between two bonded substrates, from which the stress at failure is determined. Shear bond test analyses ignore the stress distribution generated within the adherence zone, which can have a significant effect on the mode of failure. Moreover, the loading device for the shear test typically makes contact at a slight distance from the interface because of a small radius of curvature at the edge of the loading blade or wire [16]. This causes flexural failure rather than the desired shear failure. Flexural failure can lead to the false interpretation of the resultant bond strength data.

Chevron-notch specimens have been developed for testing the fracture toughness of ceramics, high-strength metals, and other brittle materials [17–22]. Barker proposed short-rod and short-bar designs for fracture toughness tests [18,19,21]. These specimens have advantages over traditional fracture toughness specimens. They are relatively small, simple, and inexpensive. Precracks are not required because there are extremely high stress concentrations at the tip of the chevron notch. Thus, cracks can be initiated at a low applied load in a stable manner. Fracture toughness can be evaluated from the maximum load. Consequently, a load-displacement record is not required and measurement of crack length is not necessary [23].

The primary advantage of the short-bar fracture toughness test is that a precrack is not needed. Other advantages include the small sample dimension, the dependence of fracture toughness on the peak load alone, freedom from measuring the crack length, and the applicability to both ductile and brittle materials. Short-rod and short-bar designs have been used to test the toughness of bonded interfaces between luting cements and dentin [24] and between resin composite and dentin [25]. However, there are no published studies that have been based on the use of the chevron-notch design for testing of bilayer, dental ceramics.

Mecholsky and Barker [26] developed a method of using the chevron-notch specimens to measure the interfacial toughness of ceramic-metal interfaces. To ensure equal compliances for the two halves of the specimens with different elastic moduli, they assumed that, $E_1 I_1 = E_2 I_2$ where $E_1 I_1$ and $E_2 I_2$ are the product of the elastic modulus and moment of inertia of materials 1 and 2, respectively. Values of d_1 and d_2 are chosen such that:

$$E_1 d_1^3 = E_2 d_2^3 \quad (1)$$

where d is the thickness of the short bar. The compliances of both halves will be approximately the same (Fig 1a).

The interfacial toughness can be derived from the equation;

$$K_c = (Y_m P_{\max} / BW^{1/2}) \quad (2)$$

Where,

K_c is the fracture toughness

Y_m is the minimum stress intensity coefficient

$P_{\max}$ is the maximum load to failure

B is the width of the specimen

W is the length of the specimen

The objective of the present study was to determine the interfacial fracture toughness of two types of bilayer dental ceramics using the short-bar chevron-notch test and to test the null hypothesis that the interfacial toughness of core-veneer bonded bilayers do not differ significantly from the fracture toughness of the monolithic veneer ceramic.

Materials and methods

Material combinations

Group LC/GV: Bilayer short-bar specimens were prepared from a lithia-disilicate glass-ceramic core bonded to a fluorapatite glass veneer, $n=16$.

Group ZC/GV: Bilayer short-bar specimens were prepared from an yttria-stabilized zirconia core bonded to a fluorapatite glass veneer, $n=16$.

Group GV/GV: Bilayer short-bar specimens were prepared from bonding fluorapatite glass-veneer bonded to itself, $n=16$.

Sample preparations—Modified T-shaped short-bar core specimens (Figs. 1b & 1c) of a lithia-disilicate-based core ceramic (LC) and an yttria-stabilized zirconia core ceramic (ZC) [6.4 mm (B) $\times$ 11.5 mm (W) $\times$ 3 mm (d_1)] were prepared using the lost-wax technique and CAD-CAM technology, respectively, according to the manufacturer's instructions (Table 1 & 2). Palladium foil with a thickness of 25 μ m was placed on the core ceramic bonding surfaces and a V-shaped notch was cut with dimensions as shown in Fig. 1c to leave the bonding area exposed (Figs. 2a & 2b). The bonding surfaces were prepared according to the manufacturer's instructions for each material. For the ZC/GV group, core ceramics (ZC) were cleaned with hot stream jet and left dry under room temperature before veneering process. ZirLiner was applied on bonding area and fired according to the recommendation prior to veneering (Table 2). For the LC/GV group, core ceramics were grit-blasted with 100 μ m Al_2O_3 particles at a pressure of 0.1 MPa and cleaned with hot stream before veneering. Veneering ceramic powder (GV) was incorporated with mixing liquid to obtain a slurry solution that was incrementally condensed on core materials and sintered in a programmable furnace (Programat P80, Ivoclar, Schaan, Liechtenstein). The thickness of veneering layers was adjusted according to equation 1. The modified T-shaped short-bar specimens (Fig. 2c) were stored in 37 °C distilled water for 30 days before testing.

For the control group, glass powder and build-up liquid were mixed and condensed in a silicone mold and sintered in a programmable furnace. The V-shaped notch and another layer of glass veneer were prepared in the same manner. The chevron-notches were prepared using palladium foil. Instead of cutting the notch into the interface, the 25- μ m-thick palladium foil was used before applying the glass veneer to create a thin, sharp notch. Because the toughness and strength of the core materials were much greater than those of the glass veneer, cut notches were formed through the veneer layers, not between both layers.

Chevron-notch specimens were tested using an Instron Universal Testing Machine (Model 5500-R) at a cross-head speed of 0.03 mm/min to promote stable crack growth until final failure occurred (Fig. 2d). The fracture surfaces were individually examined using a magnifying lens and an optical microscope to determine if the crack initiated from the tip of chevron notch. In the present study, we used $E_{ZC} = 165$ GPa, $E_{LC} = 96$ GPa and $E_{GV} = 65$ GPa (Unpublished data, Ivoclar, Vivadent AG, Schaan, Liechtenstein). Specimens whose cracks propagate away from the interface were discarded and additional specimens were made to yield 16 specimens in each group. Two specimens from the control group were discarded because cracks propagated through the bulk instead of along the interface. Figure 3 shows a fractured specimen from the LC/GV group.

To determine the fracture toughness of the glass veneer control group, eight glass veneer bars (2 mm $\times$ 4.5 mm $\times$ 28 mm) were prepared by mixing glass powder and build-up liquid, condensing in a silicone mold and sintering according to the manufacturer's instructions. Indentation cracks were produced at the middle of each bar using a Vickers indenter at a 9.8 N load. The strength of each bar was determined 24 h after indentation using a four-point flexure test with a 20.0 mm lower span and 6.7 mm upper span at a crosshead speed of 0.5 mm/min. Fracture surfaces were examined and fracture toughness of the glass veneer was calculated from the equation;

$$K_{IC} = Y\sigma c^{1/2} \quad (3)$$

where Y is a numerical constant that accounts for loading and crack geometry (1.65 for indentation cracks), c is the radius of an equivalent semicircular crack [equal to $(ab)^{1/2}$] of depth a and half-width b, and σ is the stress at failure [27].

The fracture toughness of the glass veneer was used to determine the dimensionless constant (Y_m) for the chevron-notch, short-bar specimen for the geometry used in this study. By applying the mean load-to-failure and specimen dimensions of fractured chevron-notch specimens, and the fracture toughness of the glass-veneer control group, the dimensionless constant (Y_m) was determined from the equation 2.

Statistical Analysis: The difference between the group means was analyzed using a one-way analysis of variance and *post hoc* comparison using Tukey tests was performed with an assigned α level of 0.05 (SAS for Windows 9.1.3 service pack 4, SAS Institute Inc., Cary, NC, USA.).

Results

The mean load to failure of the LC/GV Group and of the GV/GV control group were 38.9 (9.6) N and 41.9 (9.3) N, respectively, and were not significantly different ($p > 0.05$). However, the mean load to failure of the ZC/GV Group was 7.1 (4.4) N, and this was significantly less than the mean failure load of the other groups ($p < 0.05$ Table 3). The fracture toughness of the glass veneer, 0.74 (0.02) MPa-m^{1/2} was calculated using strength indentation technique. Using the fracture toughness value of the glass veneer to determine the dimensionless constant, the

interfacial toughness of the core-veneer was calculated from equation 2. The mean apparent interfacial toughness of the LC/GV group and the ZC/GV group were approximately 0.69 (0.11) and 0.13 (0.07) MPa-m^{1/2}, respectively.

Discussion

Fracture toughness represents the resistance of a material to crack propagation. This is an important property for the design of dental prostheses that are usually made from brittle materials. Because dental ceramic restorations are usually bilayer, fracture toughness of the interface is an important parameter in designing dental crowns and bridges, given that ceramic restorations often fail by chipping or delamination of the veneer layers [28,29].

Instead of calibrating specimen geometry through the measurement of crack length on the chevron notch from the compliance calibration curve [25] or through finite element analysis [30], we obtained the value of Y_m by performing a measurement on a material of known fracture toughness [18]. In this case, we used glass veneer bonded to the same glass veneer with a fracture toughness derived from the indent-strength technique as a control group to calculate the dimensionless constant (Y_m), which is approximately 12 for this specimen configuration. Furthermore, the cross-head speed at 0.03 mm/min was selected to promote stable crack growth because a fast loading rate could cause cracks to propagate unstably and the failure load would be too high. Figure 4 shows a load-displacement curve, which is typical of those from chevron-notch specimens in this study. The smooth-curve peak indicates the stable crack growth before final failure.

Shown in Figs. 5a and 5b are the SEM and optical microscopy images of the fracture surface features of LC/GV group specimens. The failures appear to initiate at the tip of the chevron notch and travel along the interface. To create a crack along the interface, the thickness of each half has to be adjusted according to equation 1 to ensure equal compliance.

In all-ceramic systems, failures have frequently been reported as chipping of the glass veneer layers. Chipping has been reported as early as the first year of service [5]. In one case, 15% of the restorations chipped within two years [11]. These failures may be caused by the weak bonding between ceramic cores and glass veneers and/or by the residual stresses developed during processing. Residual stresses are usually present in bilayer dental ceramics because of differences in the coefficients of thermal expansion/contraction (α) between the core and veneer ceramics [31]. In metal-ceramic systems, it is generally thought that a slightly greater coefficient of thermal contraction for the core ceramic compared with the glass veneer (positive mismatch) is beneficial because compressive residual stresses can be developed across the metal/veneer interface in the weaker ceramic veneer layers. However, the differences in thermal expansion or contraction coefficients should not be too great since catastrophic radial tensile stresses may also develop. Moreover, it is uncertain whether or not the positive mismatch is advantageous for all-ceramic systems. One study investigated the effect of the thermal incompatibility between ceramic core (IPS Empress) and glass veneer by using feldspathic porcelain with varying α [32]. It was found that porcelain did not show any crack when the difference of α was less than 1 ppm/K. Additionally, based on a curve fit between probability of failure and failure stress, a safe tolerance in the mismatch is less than 0.6 ppm/K for the ceramic system studied. In another study, it was claimed that a mismatch value of 0.25 ppm/K is safe, while one as high as or higher than 1 ppm/K could be dangerous to bond strength of metal-ceramic [33]. This was confirmed by three-dimension viscoelastic finite element models which predicted that all-ceramic dental prostheses with an average thermal contraction mismatch greater than ± 1.0 ppm/K will likely exhibit a relatively high percentage of failures in clinical use [34].

The study suggests that the mean interfacial toughness of the LC/GV group ($0.69 \text{ MPa}\cdot\text{m}^{1/2}$) was comparable to the apparent fracture toughness of the GV/GV control group ($0.74 \text{ MPa}\cdot\text{m}^{1/2}$), indicating sufficient bonding. However, the mean interfacial toughness of the ZC/GV group ($0.13 \text{ MPa}\cdot\text{m}^{1/2}$) was significantly lower than that of the other groups. This result suggests a need to evaluate the technique of using the same glass veneer on different types of ceramic cores under a clinical situation. This difference in the interfacial toughness may be because of the greater thermal incompatibility between the ZC and GV (1.25 ppm/K) than that of the LC/GV group (0.65 ppm/K) as reported by the manufacturer (Table 1). This incompatibility may develop unfavorable residual stresses between core and veneer ceramics in the ZC/GV group. However, these residual thermal incompatibility stresses should be further investigated. We found that the glass veneer that was hot-pressed on the zirconia core yielded a higher mean interfacial toughness than the slurry-condensed glass veneer on the zirconia core (unpublished data). Shown in Figs 6a and 6b are a photograph and a backscattered electron image of a core specimen from the ZC/GV group.

The high stress concentrations within short bar chevron-notch specimens initiated a reliable stable crack. Given that specimens have equivalent dimensions, the dimensionless constant could be determined and this technique provided a relatively reliable method for calculating interfacial fracture toughness of bilayer dental ceramics.

Conclusions

- For bilayer all-ceramic restorations with high-strength core materials, the veneering ceramics were the weakest link in the design of the structure. All-ceramic restorations often fail from chipping of veneer layers or crack initiation at the interface.
- Fracture surface examination using optical and scanning electron microscopy indicated that cracks generally propagated within the bonded interface. We conclude that the short-bar, chevron-notch test is a useful method to determine the apparent interfacial fracture toughness of bilayer dental ceramics.
- Using a material with known fracture toughness as a reference material, the dimensionless constant (Y_m) could be determined without the need for compliance calibration or finite element analysis; however, the control group specimens are necessary and the constant obtained can only be applied to a specific geometry. The calculated apparent interfacial toughness of the LC/GV group was $0.69 (0.11) \text{ MPa}\cdot\text{m}^{1/2}$ and was comparable to the apparent toughness of the control group (GV/GV). Therefore, the null hypothesis was accepted. However, the apparent toughness of the ZC/GV group was $0.13 (0.07) \text{ MPa}\cdot\text{m}^{1/2}$, which was less than the apparent toughness of the control group. Thus the null hypothesis was rejected for this bilayer ceramic group.

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Figure 1. Specimen geometry: (a) A short-bar geometry was modified for two dissimilar materials; (b) Schematic of crack plane of a specimen; and (c) Modified T-shaped short-bar specimen used in this study.

Figure 2. Specimen preparation: (a) Core ceramic before veneering; (b) V-shaped notch preparation; (c) Completed test specimen; and (d) Specimen in the test fixture.

Figure 3.
A fractured specimen of LC/GV group

Figure 4.
Load-displacement curve of a chevron-notch, short-bar specimen

Figure 5.
(a) a SEM image of fracture surface of chevron-notch specimen, and (b) optical microscopy image of a fractured surface of a chevron-notch specimen of LC/GV group

Figure 6.
A fracture surface of a chevron-notch core specimen of the ZC/GV group (a) a photograph and (b) a backscattered electron micrograph.

Table 1

Materials, ceramic type, processing temperature and coefficient of thermal expansion (α) between 100 °C and 400 °C

Ceramic Products	Ceramic Type	Processing Temperature	α (ppm/K)
IPS e.max ® Ceram (GV)	Glass veneer	Sintered at 750 °C	9.5
IPS e.max ® Press (LC)	Lithia-disilicate-based core ceramic	Hot-pressed at 930 °C	10.15
IPSe.max ® ZirCAD (ZC)	YTZP zirconia core ceramic (4–6 wt% Y ₂ O ₃)	Sintered at 1500 °C	10.75

Table 2

Dental ceramics and firing procedure used in this study (T = temperature)

Ceramics	Starting T(°C)	Heating rate	Firing T(°C)	Holding time (min)	Vacuum T on-off (°C)
GV	403	50	750	1	450–749
LC	700	60	930	25	500–930
ZC*			1500		
ZirLiner	403	60	960	4	450–959

* Specimens were fabricated by the manufacturer

Table 3

Mean failure load and the apparent toughness; groups with the same superscript are not significantly different

Ceramic groups	Number of specimens	Failure load (N)	Interfacial Toughness (MPa·m ^{1/2})
LC/GV	16	38.9 (9.6) ^a	0.69 (0.11) ^a
ZC/GV	16	7.1 (4.4) ^b	0.13 (0.07) ^b
GV/GV	16	41.9 (9.6) ^a	0.74 (0.17) ^a