

## **Bonding measurement —Strength and fracture mechanics approaches**

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This study investigated the effect of cross-sectional areas on interfacial fracture toughness and bond strength of bilayered dental ceramics. Zirconia core ceramics were veneered and cut to produce specimens with three different cross-sectional areas. Additionally, monolithic specimens of glass veneer were also prepared. The specimens were tested in tension until fracture at the interface and reported as bond strength. Fracture surfaces were observed, and the apparent interfacial toughness was determined from critical crack size and failure stress. The results showed that cross-sectional area had no effect on the interfacial toughness whereas such factor had a significant effect on interfacial bond strength. The study revealed that cross-sectional area had no effect on the interfacial toughness, but had a significant effect on interfacial bond strength. The interfacial toughness may be a more reliable indicator for interfacial bond quality than interfacial bond strength.

**Keywords:** Interfacial toughness, Fractography, Microtensile, Zirconia

### INTRODUCTION

Esthetic dental restorations have long been used with success. Introduced in the late 1940s, porcelain-fused to-metal (PFM) fixed dental prostheses (FDPs) have been continued to be the most common complete-coverage anterior restorations. This type of restoration has lessened in popularity due to esthetics, biocompatibility, and a concern of metals. Recently, the newly developed high-strength core ceramics, such as zirconia and alumina were introduced as alternatives for metals. Zirconium dioxide or zirconia ( $ZrO_2$ ) has a flexural strength similar to that of steel, and it has been used as an alternative to metal for substructure of fixed dental prostheses. Nevertheless, high strength cores are used to support more esthetic, more translucent, but weaker glass veneers. In addition, since all-ceramic restorations are usually fabricated as bilayered structures, failures were frequently reported as delamination or chipping of the glass veneer as a risk of using these materials. An increasing number of clinical failures have been reported as the crack extension in the veneering glass<sup>1-6</sup>.

Shear and microtensile bond tests have been used to investigate the bond strength of two substrates, however, shear bond test creates the fracture away from the interface because of the nonuniform stress distribution<sup>7</sup> and specimen geometry<sup>8</sup>. Microtensile bond test first developed to determine the bond strength between tooth structure and adhesive materials<sup>9</sup>, and was also used to test bond strength of core-veneer ceramics<sup>10</sup>. Microtensile test produces relatively uniform stress distribution. However, most of the tests did not identify the failure origins or all the failure modes were included in the bond strength value. When the interfacial strength is comparable to the cohesive strength of the

substrates, the failures tend to appear in the weaker substrate because of the higher probability of finding critical flaws in substrate due to the larger volume of the substrate than that of the interface. Moreover, the microtensile bond strength value is inversely related to the surface area of test specimens<sup>11-14</sup>.

Veneering techniques either slurry-condensed (SC) or hot-pressing (HP) techniques have been studied and developed aiming to obtain the ultimate bond strength with zirconia substructure. Recent studies tried to overcome limitations of manual layering, such as voids or defects from nonhomogeneous material condensation, and therefore enhanced the core-veneer bond strength of zirconia restoration with hot-pressing technology<sup>1,10,15-17</sup>. However, the depth of layered esthetics may be limited when pressable glass veneer was used because of the one-shade ingot<sup>18</sup>.

Concept of fracture toughness is generally accepted as an appropriate method comparing with the strength approach to determine the mechanical property. Because the strength value can vary with specimen dimensions and flaw distribution, the value will be higher if the smaller sizes of specimens are investigated. This is because the higher probability of finding the critical flaw in the larger specimens. The fracture toughness is assumed to be constant regardless of specimen dimension.

Fractographic analysis and fracture mechanics had been applied to dentistry in order to study experimentally and/or clinically failed specimens<sup>19,20</sup>. Ceramics are prone to fracture that occurs with limited or no plastic deformation. Fortunately, their fracture patterns and fracture surface markings provide usefully interpretable information. An important step in fractographic analysis is pattern recognition. Fracture leaves telltale fracture patterns on the fracture surfaces. The existence of four distinct regions surrounding the failure initiation site

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in brittle materials, showed in Fig. 1, are: 1) the mirror region, 2) the mist region, 3) the hackle region, and 4) the macroscopic crack branching region, which is the bifurcation of the main crack. These patterns lead observer back to the origin of fracture. The fracture toughness ( $K_{Ic}$ ) of a material represents the resistance of a material to rapid crack propagation, and it was determined from the critical flaw size of the fracture surface and fracture strength using the equation:

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

Where, Y is a numerical constant that accounts for loading and crack geometry. The value of the constant (Y) is equal 1.24 for the surface crack<sup>20</sup>, 1.4 for the corner crack<sup>21</sup>, and 1.3 for the inner crack<sup>22</sup>.  $\sigma$  is the failure stress, and c is the radius of an equivalent semicircular critical crack. c can be calculated from:

$$c = (ab)^{1/2} \quad (2)$$

where a is depth and b is half-width of the critical crack (Fig. 1). If the critical crack and stress at failure can be determined, the value of the fracture toughness can also be calculated.

Microtensile bond test was created in order to produce a relatively uniform stress distribution as it minimized the influence of interfacial defects because of a small bonding area, resulting in a more accurate

evaluation of the bond strength<sup>15</sup>. Subsequently, it has also been used to test the bond strength of core/veneer ceramic systems<sup>10,15-17,23</sup>. The core/veneer interfacial bond quality plays an important role in the mechanical properties of layered restorations. The microtensile bond strength test is a useful method to study bond quality of the bilayered materials when subjected to tensile stresses. However, the obtained stresses may not represent the actual interfacial bond strength since a failures were generally included in the analysis even though some of those might have failed cohesively. In addition, the microtensile bond strength was reported to be inversely related to the cross-sectional area of the test specimens<sup>11-14</sup>. Since the bond strength can vary with specimen dimensions and flaw distributions, a report of higher bond strength in a small-sized specimen can be misleading if one compares it with a relatively large specimen. None of the test procedures used so far is able to evaluate the actual value of bond strengths for any material and neither is it possible to compare data from one researcher to another<sup>8</sup>.

The objective of the study was to test the hypothesis that (1) There is no effect of cross-sectional areas on the interfacial fracture toughness and bond strength of bilayered dental ceramics, (2) There is no effect of cross-sectional area on the apparent toughness and tensile strength of monolithic glass veneer.

## MATERIALS AND METHODS

Yttria-stabilized zirconia core ceramics (IPS e.max ZirCAD) were prepared and sintered as blocks with dimension of 10×10×5 mm. After sintering, the zirconia blocks were then veneered with two different veneering techniques; a hot-pressing technique [HP] (IPS e.max® ZirPress) and a slurry-condensed technique [SC] (IPS e.max® Ceram), according to manufacturer recommendation. The bonding surfaces of the zirconia blocks were cleaned thoroughly with a steam jet and air dry. Subsequently a thin layer of ZirLiner (IPS e.max® Ceram ZirLiner, Ivoclar Vivadent, Schaan, Lichtenstein) was applied and fired prior to each veneering procedure. Zirconia blocks with ZirLiner were fired in the furnace according to the manufacturer instruction. Materials, composition, and processing temperatures for both veneering techniques were listed in Table 1. Once the ceramic block with ZirLiner was completely sintered and left cool down to room temperature, veneering ceramic (IPS e.max® Ceram) was mixed to a creamy consistency and applied on a zirconia

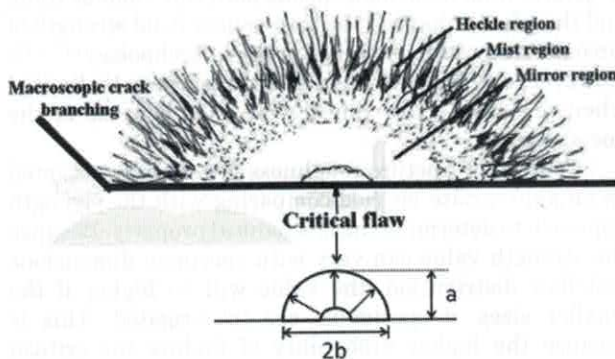


Fig. 1 Schematics of four distinct regions surrounding the failure initiation site in brittle materials; critical flaw, the mirror region, the mist region, the hackle region, and the macroscopic crack branching region.

Table 1 Materials, composition and processing temperature of materials used in this study

Material	Composition	Processing temperature (°C)
IPS e.max® ZirCAD (ZC)	Y-TZP zirconia core ceramic (4–6 wt%Y <sub>2</sub> O <sub>3</sub> )	Sintering at 1,500
IPS e.max® ZirPress (HP)	Fluorapatite glass	Hot pressing at 910
IPS e.max® Ceram (SC)	Low-fusing fluorapatite glass	Sintering at 760

core for SC technique. Once removal of excess water and condensation were performed, the blocks were then sintered according to manufacturer's recommendation. After the blocks cooled, other increments were applied to gain approximately 2.5 mm thick layer.

For HP technique, ZirLiner was applied and sintered on zirconia, the same as SC technique. Subsequently, modeling wax was added to a thickness of 2.5 mm. The blocks were sprued and put in the investing silicone ring. Once the investment was completely set, it was put in the furnace in order to burn the wax away. ZirPress (IPS e.max® ZirPress) ingots were placed, followed by the plunger into the access hole. Once firing cycle was complete, investment was removed by blasting with glass beads followed by Al<sub>2</sub>O<sub>3</sub> particles. Subsequently, the sprues were cut and bilayered ceramic blocks were ready for cutting process.

After veneering processes, bilayered ceramic blocks were serially cut with a low-speed diamond saw (IsoMet® Lowspeed saw, Buehler, IL, USA) resulting in microtensile test bars with cross section of 1×1, 1.2×1.2, and 1.5×1.5 mm<sup>2</sup> which were equivalent to cross-sectional areas of 1.0, 1.44, and 2.25 mm<sup>2</sup>, respectively, for both HP and SC groups. Specimens from the external border of the block were discarded to avoid the effect of surface stresses induced by polishing and thermal contraction. Test specimens finally had equal length of core and veneer ceramics, which was approximately 2.5 mm each. Microtensile bar specimen preparation was showed in Fig. 2.

Additionally, microtensile tested bar were also prepared from monolithic glass veneer (IPS e.max® ZirPress) as a control group. The resin blocks of 10×10×6 mm were prepared by mixing powder and liquid of the pattern resin (Duralay, Reliance Dental Mfg., Alsip, IL, USA) and poured into a silicone mold. The resin blocks were invested and burnt out in the furnace. The IPS e.max® ZirPress ingot was heat-pressed into the investment ring to achieve the monolithic ceramic blocks, as described in HP technique. Finally, the monolithic microtensile test specimens (*n*=16) with

three different cross-sectional areas of 1.0, 1.44, and 2.25 mm<sup>2</sup>, respectively were obtained from the cutting, same as the bilayered blocks.

The specimen was attached to the flat grips using cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan) and tested in tension until fracture using the universal testing machine (Instron 5566, Instron, Buckinghamshire, England) at a crosshead speed of 0.5 mm/min. Microtensile bar specimens were randomly selected from each group and tested to yield 16 specimens that developed interfacial fracture (*n*=16). Interfacial bond strengths for specimens with different types of veneering materials (HP or CS) and with different cross-sectional areas (1.0, 1.44, or 2.25 mm<sup>2</sup>) were recorded. The monolithic ZirPress specimens were also tested in tension until fractured and recorded as tensile strength of the material. The specimen fragments were transferred and stored individually to minimize the abrasion and other surface damage to the fracture surfaces.

Fracture surfaces were observed with an optical microscope (Nikon eclipse E400 POL, Nikon, Tokyo, Japan) at 100× and 200× magnifications to find the fracture origin. Critical crack size images were captured by a digital camera and measured by image analysis software (Image-Pro Plus version 3.0, Media Cybernetics, Rockville, MD, USA). A calibration glass slide was used to calibrate the measurement at certain magnifications. In addition, scanning electron microscope (JSM-5410LV, JEOL, Tokyo, Japan) was also used to verify the location of a critical crack in complicated specimens because of the greater depth of field.

The apparent toughness was also calculated for specimens with different types of veneering materials (HP or SC) and monolithic ZirPress, and with different cross-sectional areas (1.0, 1.44, or 2.25 mm<sup>2</sup>). Mean apparent toughness of the interface and mean bond strength of HP and SC groups with varying cross-sectional areas were statistically tested with One-way ANOVA and Tukey HSD at 95 percent confident interval (*p*<0.05).

## RESULTS

Figure 3 shows the SEM micrograph of the fracture surface and a critical crack of a microtensile test specimen. The mean interfacial bond strength, mean interfacial toughness, and standard deviation (SD) for different types of veneering techniques and different cross-sectional areas of bilayered dental ceramics are shown in Table 2. Sixteen specimens were selected where failures developed at the interface. In addition, the tensile strength and apparent toughness of a glass veneer (control group) is shown in Table 3.

One-way ANOVA and Tukey HSD revealed that cross-sectional area had no effect on the interfacial toughness (*p*>0.05) whereas such factor had a significant effect on interfacial bond strength (*p*<0.05) in both ceramic groups. The smaller cross sectional areas had greater bond strength value than those of larger cross-

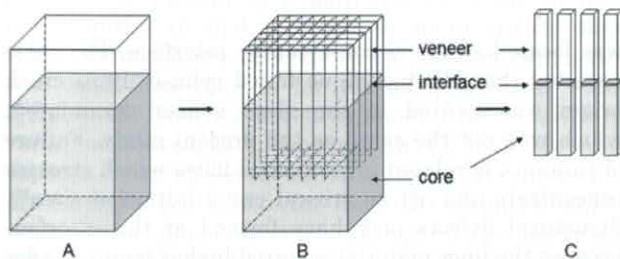


Fig. 2 Schematics of microtensile bar specimen preparation; (A) zirconia core ceramic block was veneered with either HP or CS techniques (B) Bilayered ceramic block was serially cut into desired shape. Specimens from the external border were discarded, (C) Final bilayered microtensile bar specimen.

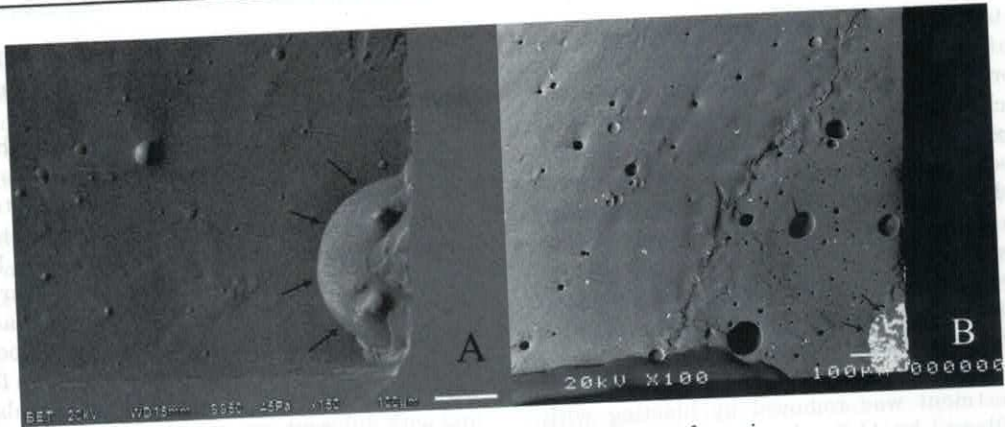


Fig. 3 Schematics of a fracture surface of a microtensile tested specimen. Arrows demonstrate a critical crack boundary (A) surface crack of IPS e.max® Ceram monolithic glass veneer specimen (B) corner crack of bilayered IPS e.max® ZirCAD/IPS e.max® Ceram (zirconia side).

Table 2 Mean interfacial bond strength and mean interfacial toughness (SD in parenthesis) of two veneering techniques and three different cross-sectional areas of bilayered dental ceramics

Veneering techniques	Interfacial bond strength (MPa)			Interfacial toughness (MPa · m <sup>1/2</sup> )		
	1.0 mm <sup>2</sup>	1.44 mm <sup>2</sup>	2.25 mm <sup>2</sup>	1.0 mm <sup>2</sup>	1.44 mm <sup>2</sup>	2.25 mm <sup>2</sup>
SC	21.2 [3.2] <sup>A</sup>	16.8 [3.8] <sup>B</sup>	12.7 [2.9] <sup>C</sup>	0.32 (0.05)	0.32 (0.05)	0.27 (0.06)
HP	17.6 [6.1] <sup>a</sup>	14.8 [5.1] <sup>ab</sup>	12.0 [3.1] <sup>c</sup>	0.23 (0.08)	0.21 (0.05)	0.19 (0.06)

The values with the same superscript are not significantly different.

Table 3 Mean tensile strength and mean apparent toughness (SD in parenthesis) of monolithic glass veneer (IPS e.max® ZirPress) with three different cross-sectional areas

IPS e.max® ZirPress	Tensile strength (MPa)			Apparent toughness (MPa · m <sup>1/2</sup> )		
	1.0 mm <sup>2</sup>	1.44 mm <sup>2</sup>	2.25 mm <sup>2</sup>	1.0 mm <sup>2</sup>	1.44 mm <sup>2</sup>	2.25 mm <sup>2</sup>
	26.9 [5.6] <sup>a</sup>	16.8 [3.8] <sup>ab</sup>	12.7 [2.9] <sup>b</sup>	0.32 [0.05]	0.32 [0.05]	0.28 [0.06]

The values with the same superscript are not significantly different.

sectional areas. For the control group, it was also found that there were significant differences of tensile strength between monolithic glass veneers with different cross-sectional areas. However, no effect of the cross-sectional areas was found in the apparent toughness value of monolithic glass veneer.

#### DISCUSSION

In this study, fracture primarily occurred near the interface of core/veneer with residual veneer remaining on the core, and no evidence of fracture occurred in the core material. This was partly in agreement with a previous research in that all zirconia frameworks remained intact and the crack origin for SC technique

was located at the zirconia-veneer interface. They also reported that HP technique failed cohesively as crack origin was located in the glass veneer ceramic<sup>10,15</sup>, which was not the same as the present study. Failure of ceramics is related to structural flaws which stresses concentrate and act as critical crack initiation sites<sup>24</sup>. Structural defects may have formed at the interface because the liner material required higher liquid-powder ratio relative to the consistency of normal veneering slurry. Other defects may be caused by microspaces in the glass phase of the veneer due to transformation from tetragonal to monolithic phase of zirconium oxide at the core-veneer interface which is accompanied by generating localized stress<sup>25</sup>. In a study, more than 90 percent of their failure mode was interfacial failure<sup>23</sup>,

which was similar to this study. It was found that for zirconia-glass veneer combinations, the low thermal diffusivity of the zirconia resulted in the highest temperature difference between framework and veneering porcelain causing high residual stresses at the interface<sup>26</sup>. The residual stress in bilayered ceramics is primarily relative to the thermal expansion mismatch of the two materials. The differences in coefficient of thermal expansion (CTE) resulted in cracking, depended on whether the glass veneer has a higher or lower CTE compared to that of core<sup>27</sup>. The thermal expansion of the glass veneer is generally slightly lower than that of the framework or core materials in order to create circumferential compressive stresses at the glass veneer interface with compensating tensile stresses at the interface of the core. The coefficient of thermal expansion (CTE) of the core and the veneering materials may cause the result of bond strength between HP and SC techniques to be not different. When the CTE of core/veneer is much different, residual stresses are usually present<sup>26,28,29</sup>. The magnitude of the residual stresses in the glass veneer is directly related to the thermal expansion mismatch between coping and veneering material<sup>26</sup>. Steiner *et al.* investigated the effect of thermal mismatch between IPS Empress ceramic core and glass veneers with varying CTE. It was found that veneers did not develop crack if the mismatch was less than 1 ppm/K<sup>30</sup>. Another study claimed that a mismatch value of 0.25 ppm/K was considered safe while one with mismatch higher than 1 ppm/K could be dangerous to bond strength of core/veneer<sup>31</sup>. As reported by manufacturer's data sheets, CTE (100–400°C) of IPS e.max® ZirCAD, IPS e.max® ZirPress, and IPSe.max® Ceram were 10.8, 9.8, and 9.5 ppm/K, respectively. Thus core/veneer thermal contraction mismatch between ZirCAD/ZirPress and ZirCAD/Ceram were quite similar (1 and 1.3 ppm/K, respectively) which were considered high for bond strength beyond the safe zone. This was confirmed by finite element models which predicted that CTE mismatch of all-ceramic prostheses greater than 1 ppm/K will showed high likelihood of failures<sup>32</sup>.

The fracture strength of brittle materials such as ceramics is not generally reproducible. As flaws are already present in a material, there are greater opportunities to find a larger or more critical flaw while testing larger specimens. In addition, when the test conditions change, for example, stressing rate, temperature, and testing environment, those result in variation of strength<sup>33</sup>. The strength of a given material depends on the size of the initiating crack present during processing in contrast to the fracture toughness, which is generally independent of the size of the initiating crack<sup>20</sup>. In this study, it showed that interfacial bond strength was sensitive to cross-sectional area of the specimens which was agreed by many researchers that it was inversely related to the cross-sectional area of the test specimens<sup>11-14</sup>. Moreover, average stress underestimated the actual bond strength at a particular part of the component<sup>7,8</sup>. The core/veneer bond strength and interfacial quality played an important factor that

could significantly influence the mechanical properties of layered restorations. Most of the previous tests did not use fractography approach to identify the failure origin. All failures had been generally included in the analysis of bond strength even though some of those have failed cohesively. When the interfacial strength is comparable to the cohesive strength of the substrates, the failures tend to appear in the substrate because of the higher probability of finding critical flaws in the larger volume of the substrate rather than those found along the interface<sup>33</sup>. Since the bond strength can vary with specimen dimensions and flaw distributions, a report of higher bond strength in a small-sized specimen can be misleading if one compares it with a relatively larger specimen. Furthermore, when the bonding between two materials is comparatively high, failure at the interface rarely occurs as we found in our pilot study, therefore, the result of traditional bond strength tests may be not valid in high bond strength materials.

This study showed that cross-sectional areas had no effect on interfacial toughness. Interfacial toughness should be independent of specimen dimensions and was more reliable than bond strength for determining the bond quality between core and veneer ceramics. It might be a more appropriate approach to determine bonding quality. However, there were limited studies that used this technique to determine the interfacial toughness of bonded bilayered ceramic structures. We believed that this concept could be applied to other varieties of brittle bilayered materials as well. In recent studies, fractography had been applied to dentine<sup>34</sup>, indirect dental resin composites<sup>35</sup>, and even ornamental stones<sup>36</sup>. They also recommended that toughness approach should possibly be used to compare studies instead of strength since toughness values are intrinsic to the materials<sup>34,35</sup>. Recently, many studies have applied fracture mechanics concept to the tests and recommended that they were more appropriate approaches to determine the bonding quality between two materials compared with traditional tests such as microtensile or shear bond tests<sup>37-41</sup>.

There should be further studies on the interfacial toughness to compare the reliability of this technique with other different test methods and on many types of materials and products. However, none of the available techniques were proved to be straightforward. Selection of the test methods should be based on a thorough understanding of the advantages, disadvantages, technical difficulties, and limitations inherent to each technique.

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