Original Article

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Fracture mechanics approach to determine bonding quality of two ceramics

Vachiraporn Umpornsittikul[1], Chuchai Anunmana[1], Potchaman Sinavarat[1], Pornkiat Churnjitapirom[2]

[1] *Department of Prosthodontics, Faculty of Dentistry, Mahidol University*
[2] *Research office, Faculty of Dentistry, Mahidol University*

Objective: To evaluate bonding quality of bilayered and monolithic dental ceramics using chevron-notch specimens.
Materials and methods: The monolithic blocks of ceramic core materials (IPSe.max®Press) and ceramic veneering material (IPSe.max®Ceram) were fabricated. In addition, the blocks of bilayered ceramic (IPS e.max®Press/IPSe.max®Ceram) was also fabricated. The triangular notch shape were created at the middle of the prepared bars and at the interface between two ceramics (3x4x16 mm) by using the specific devices and loaded in tension until failure. Optical microscope and Scanning Electron Microscope (SEM) were used to determine the crack propagation and ensure the mode of failure. Then, the bonding quality of the bilayered and monolithic specimens were analyzed.
Results: All of the specimen failures were found at the interface. The optical microscope revealed that the crack was initiated at the tip of the triangular notch and the failure occurred along the interface. Accordingly, the interfacial failure pattern was classified.
Conclusions: The test reveals the different approach to evaluate the bonding effectiveness of the ceramics. Specimen size and cutting angle of the triangular notch can be control. The sample can be readily prepared. Therefore, this test can be used as a valid alternative to analysed the bonding quality of the ceramics because the approach ensured the interfacial failure.

Key words: all ceramic, bilayered, bonding, chevron-notch, fracture toughness, interfacial

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Introduction

All-ceramic restorations have been widely used as fixed prostheses due to their superior esthetics and excellent biocompatibility. Several types of ceramic are commercially available such as lithium disilicate, zirconia, alumina and their matching veneering materials. The high strength ceramic core materials are used as alternatives to metal. However, failures of all-ceramic restorations were fracture of the core materials, chipping and fracture of veneering layers [1,2].

The bond between ceramic core material and the corresponding veneering material might be one of the weakness that can affect the long-term success of all-ceramic restoration [3-5]. The strength of the core-veneer bond is influenced by the residual stresses developed from the CTE mismatch or from the rapid or slow cooling rate during firing [6-8]. The difference in elastic modulus of ceramics core and veneering material is also the affecting factor [9,10]. To evaluate the effectiveness of the ceramic/ceramic bonding, shear bond test, tensile bond test, micro shear

Corresponding author: Vachiraporn Umpornsittikul, Department of Prosthodontics, Faculty of Dentistry, Mahidol University, 6 Yothi Road, Ratchathevi, Bangkok 10400, Thailand Tel: +66-2-203-6440 E-mail: peachdentmu@gmail.com
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bond test (μSBS) and micro tensile bond test (μTBS) have been used [11-13]. However, the results presented the variability of the fracture pattern, mostly found cohesively in the veneering layers [12]. This means that it represents the strength of the veneering material instead of the bond strength of the core-veneer interface.

In order to obtain an appropriate bonding test method, the chevron-notch test has been developed for testing the interfacial fracture toughness of ceramics or other brittle materials [14-17]. This method provides some advantages. The pre-crack is not needed because high stress concentration presents at the tip of chevron-notch. The tip of chevron-notch, which is the weakest part, can cause the crack at the low applied loads. Fracture toughness can be easily calculated from the maximum test loads and calibration factor, depending on specimen geometry and loading configuration [18,19]. Moreover, it uses small, simple dimension and inexpensive [20]. Consequently, the measurement of fracture resistant at the interface is possible.

The objective of this study is to evaluate bonding quality of bilayered and monolithic dental ceramics using chevron-notch specimens.

Materials and methods

Three groups of specimens were established with 15 specimens per group.

- Group 1 : IPS e.max® Ceram (monolithic)
- Group 2 : IPS e.max® Press (monolithic)
- Group 3:IPS e.max® Press/ IPS e.max®Ceram (bilayered)

Preparation of monolithic specimens

Dentine porcelain powder (IPS e.max® Ceram) and liquid were mixed and condensed into plastic mold. The first layer was fired according to manufacturer's instructions. The additional layers were added with the same technique to gain the monolithic blocks with final dimension of 3 mm x 4 mm x 16 mm.

For IPS e.max® Press, resin blocks (3 mm x 4 mm x 16 mm) were prepared. After that, the lost-wax technique was done to gain the final dimension of the monolithic specimens (3 mm x 4 mm x 16 mm).

Preparation of bilayered lithium disilicate core ceramics-veneer specimens.

Resin blocks (3 mm x 4 mm x 8 mm) were prepared, sprued and invested according to manufacturer's instruction. Then, the investing process was completed. The completed lithium disilicate blocks were placed in plastic mold for veneering process. IPS e.max® Ceram dentine porcelain powder and the modeling liquid were mixed and condensed on the IPS e.max® Press block, which placed in plastics mold using load transferring device with 10 kg load and the load was maintained for 5 minutes. The three cycles of veneering were done and fired according to manufacturer's instruction to achieve the bilayered specimens. Finally, the specimens were finish and polished to derive the specimens with the dimension of 3 mm x 4 mm x 16 mm (Figure 1).

Cutting process

The prepared bilayers and monolithic blocks were cut in to triangular notch shape (3 mm x 3 mm x 3 mm.) at the interfacial zone or at the middle of the monolithic blocks using a low-speed diamond saw (Isomet, Buehler, Illinois, USA) under water cooling. The specimen was secured using a holding device to ensure that all the specimens were prepared with the same cutting angle (Figure 2).

The finished specimens were tested in

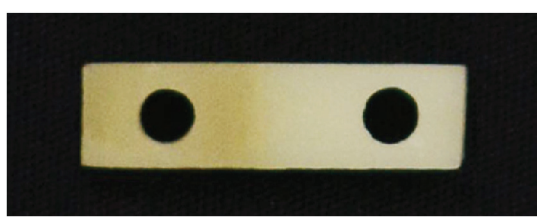

Figure 1. IPS e.max® Press/IPS e.max® Ceram bilayered specimens

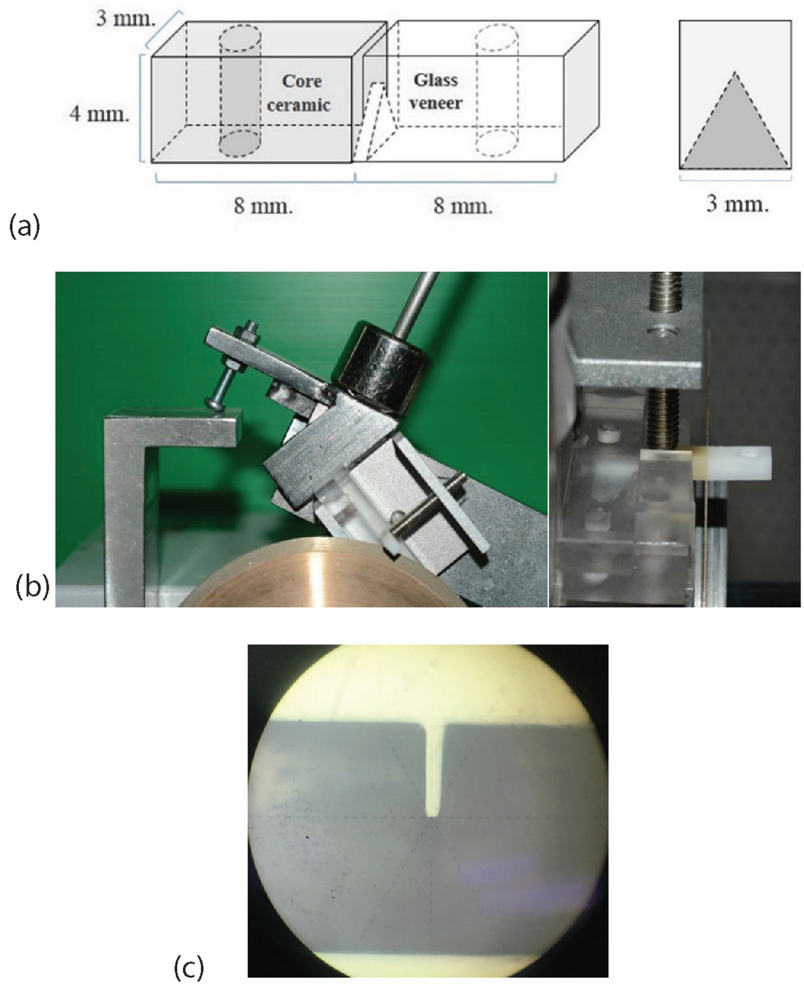

Figure 2. Cutting process
(a) chevron-notch bar specimens geometry
(b) Preparation of specimens with the same cutting angle
(c) Prepared specimens after cutting process

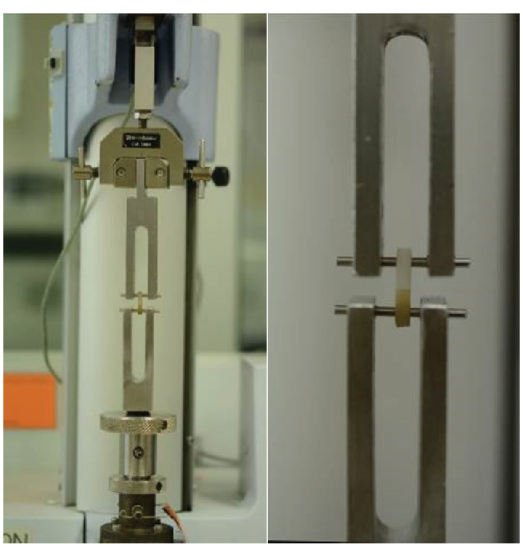

Figure 3. Specimens were loaded in tension to failure.

tension at a crosshead speed of 0.1 mm/min using a universal testing machine (EZ-S, Shimadzu, Japan). Specimen was tightened on stainless steel wire at the circular notch and attached to the grips of testing machine. Specimen was paralleled to the long axis of the device to avoid bending stress. After that, the samples were loaded until failure occurred (Figure 3).

The loads at failure were recorded and calculated using the equation.

$$K_{IC} = \frac{P_{max} \times Y_m}{W \times L^{1/2}}$$

K_{IC} is the apparent fracture toughness

Y_m is the minimum stress intensity coefficient
P_{max} is the maximum load to failure
W is the width of the specimen
L is the length of the specimen

Results

The mean and standard deviation of apparent fracture toughness were summarized in Table 1. Data analysis was performed using One-way ANOVA and Dunnett's test at a significant level of 0.05.

The mean apparent fracture toughness of the monolithic IPS e.max® Ceram was significantly greater than that of the bilayered IPS e.max® Press/IPS e.max® Ceram, whereas, the apparent fracture toughness of IPS e.max® Press was significantly greater than those of the other groups.

After testing, specimens were observed using magnifying glass and optical microscope to determine if the crack was initiated at the tip of chevron-notch and the failure occurred along the interface. All of the specimen failures were found at the prepared triangular notch. The optical microscope revealed that the crack was initiated at the tip of the triangular notch and the failure occurred along the interface (Figure 4).

The fracture patterns were examined under SEM to ensure the modes of failure. It was found that the fracture originated between core ceramics (IPS e.max®Press) and veneering half (IPS e.max® Ceram). Accordingly, the interfacial failure pattern was classified (Figure 5 & 6).

Discussion

Microtensile bond strength test is one of the most practical test methods to study the bond strength of core-veneer ceramic restorations. However, the stresses obtained from these studies might not represent the actual bond strength since the failure did not occur at the interface [12]. Moreover, micro tensile bond strength is influenced by specimen geometry, substrates and thickness of the substrates [16]. Chevron Notch Beam (CNB) was developed and used to evaluate the interfacial fracture toughness [21,22]. This test was first developed for fracture toughness test of high strength metal or ceramics, and other brittle materials [23,24]. Anunmana et al. [20] used this test to investigate the interfacial fracture toughness of bonded core-veneer bilayered dental ceramics by initiating the crack propagation through the bonded interface of the core ceramics and their corresponding veneer using the chevron-notch short bars.

In this study, the bonded interface was modified from the previous study [20] to be 3x4mm in order to obtain smaller specimens and to decrease the internal defects. It was much simpler for this specimen geometry and no specimens failed during preparation. According to the effects of surface bonding area, the smaller bonding area produced the higher bond strength than the larger bonding area [24]. Although the preparation of the

Table 1. Mean and standard deviation of apparent toughness ($MPam^{1/2}$)

Ceramic groups	Number of specimens	Interfacial toughness ($MPam^{1/2}$)
IPS e.max® Ceram	15	0.75 ± 0.06^a
IPS e.max® Press	15	2.34 ± 0.28^b
IPS e.max® Press/IPS e.max® Ceram	15	0.68 ± 0.05^c

Groups with the same superscript are not significantly different.

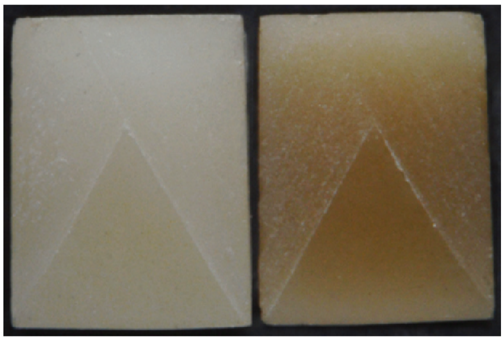

Figure 4. The representative image of fractured specimens of IPS e.max®Press/IPSe.max® Ceram. The ceramic core materials were on the left side and their corresponding glass veneers were on the right. Failure pattern was classified as interfacial failure.

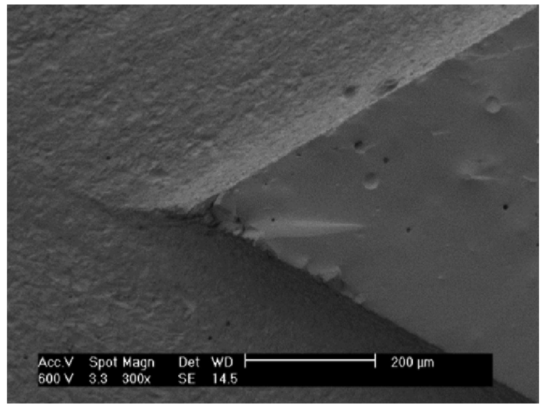

Figure 5. The representative SEM image of fractured surface of monolithic blocks of glass veneer (IPS e.max®Ceram) reveal critical crack of specimen that originated from the tip of chevron-notch.

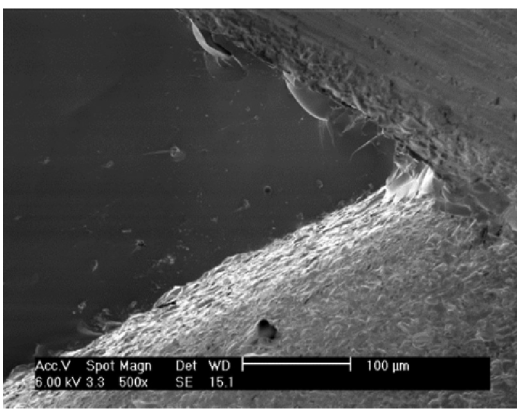

Figure 6. The representative SEM image of fractured surface of core-veneer (IPS e.max®Press/IPS e.max®Ceram) showed different surface between exposed surface of core material and veneering side, indicating the interfacial failure.

chevron-notch bars require a specific technique and devices, the sample preparation could be carefully prepared. The tensile mode was used to avoid the bending stress. The slow loading rate was suggested to propagate the stable crack growth. Therefore, the crosshead speed 0.1 mm/min was chosen. The load-displacement curve of a specimen tested in this study is shown in (Figure 7).

The curve peak represented a stable crack growth, which typically obtained from the chevron-notch specimens in this study. After the test started, there was a non-linear increasing load pattern, then, it turned into the linear mode. After that, the slope of loading curve was decreased to almost zero which was attributed to the stable

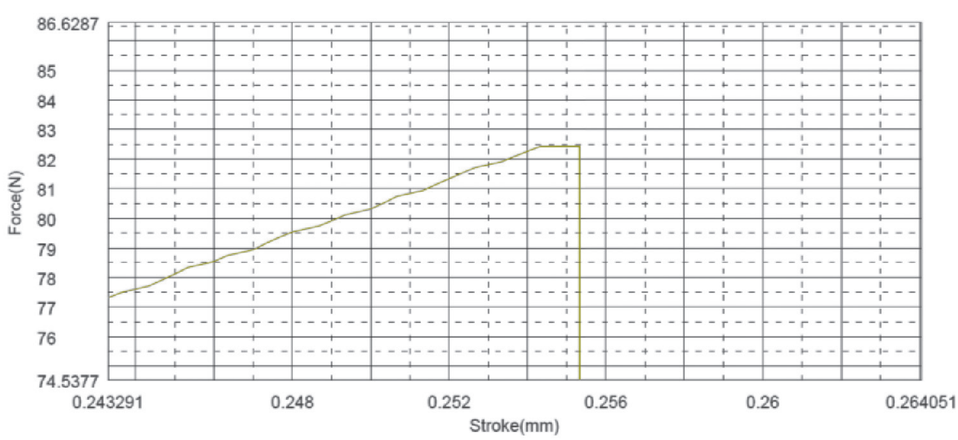

Figure 7. The load-displacement of a chevron-notch specimens.

crack growth.

In calculation of the apparent interfacial fracture toughness, the value of Y_m was calibrated from the material which known fracture toughness. Based on a previous study [20], the monolithic glass veneer was used as a control group to calculate the Y_m constant. It was approximately 5 for these specimens. We recognized the triangular notch as the one of the weakest part since all of the specimens fracture occurred at the interfacial area. The pre-crack loads were not required due to the high stress concentration propagated at the tip of the chevron notch. Correlatively to the studies in 2016 [25,26], the short rod chevron-notch was used to assess the enamel bonding effectiveness. The overall failure of specimens propagated through the interface. Moreover, the finite element analysis model revealed the mainly tensile stress found at the tip of the triangular notch. Likewise, all specimens in this study fracture at the prepared triangular notch when they were observed using a stereomicroscope. The crack propagated between or near the core-veneer layer. Correspondingly, the interfacial fracture toughness value of the bilayered group IPS e.max® Press/IPS e.max® Ceram was significantly lower than the fracture toughness of the monolithic veneer IPS e.max® Ceram. This study suggested that the fracture toughness of the interface was not greater than the fracture toughness of the veneering material.

The failures of bilayer ceramic restoration might occur due to the residual stresses. Taskonak et al. [7] reported that the residual stress values were detected in bilayer lithium disilicate core-veneer specimen. The incompatibilities of CTE cause the residual stress to develop between core and veneering ceramics [8,27]. Mismatch of CTE also generates stress gradients within the veneer and affects the crack propagation, likewise, it affects the interfacial adhesion between core ceramics and their veneer layers. Although, the previous studies [6,10] suggested that the minimal difference in CTE can increase the chipping resistance of bilayered ceramic restorations in the clinical application, Dehoff et al. [9] suggested that the CTE difference in the range of $-0.61\times10^{-6}\cdot K^{-1}$ to $+1.02\times10^{-6}\cdot K^{-1}$ is acceptable. Base on the present result in this study, the chevron-notch test can be used as an alternative test to determine the apparent interfacial fracture toughness of bilayered dental ceramics. However, in preparation of the triangular notch at the interface using the diamond blade might cause the microcracks near the cutting area and these cracks might interfere with the crack propagation during testing. Further improvement in preparation of triangular notch at the interface such as a gentle cut using the laser technology should be used if possible. The size of the specimen needs to be verified. The further studies in different type of ceramics are useful. In addition, the study about stress distribution between core and veneer of bilayered specimens may be useful in explaining the fracture propagation and pattern for this test.

Conclusion

In this study, the chevron-notch approach was used to evaluate the effectiveness of the ceramic/ceramic bonding. Fracture surface examination indicated the crack propagated within the interface. Specimen size and cutting angle of the triangular notch could be controlled. Therefore, this test can be used as a valid alternative to analyse the bonding quality of the adjoining ceramic materials.

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Ethical approval : No requirement

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