INFLUENCE OF INVESTMENT/CERAMIC INTERACTION LAYER ON INTERFACIAL TOUGHNESS OF BODY CERAMIC BONDED TO LITHIA-BASED CERAMIC

Ju-Mi Park, D.D.S., M.S.D., Ph.D.
Department of Prosthodontics, School of Dentistry, Chonbuk National University, Korea

Statement of problem. Interfacial toughness is important in the mechanical property of layered dental ceramics such as core-veneered all-ceramic dental materials. The interfaces between adjacent layers must be strongly bonded to prevent delamination, however the weak interface makes delamination by the growth of lateral cracks along the interface.

Purpose. The purpose of this study was to determine the effect of the reaction layer on the interfacial fracture toughness of the core / veneer structure according to the five different divesting.

Materials and methods. Thirty five heat-pressed Lithia-based ceramic core bars (IPS Empress 2), 20mm × 3mm × 2mm were made following the five different surface divesting conditions. G1 was no dissolution or sandblasting of the interaction layer. G2 and G3 were dissolved layer with 0.2% HF in an ultrasonic unit for 15min and 30 min. G4 and G5 were dissolved layer for 15min and 30min and then same sandblasting for 60s each. We veneered bilayered ceramic bars, 20mm × 2.8mm × 3.8mm(2mm core and 1.8mm veneer), according to the manufacturer' s instruction. After polishing the specimens through 1 ㎛ alumina, we induced five cracks for each of five groups within the veneer close to interface under an applied indenter load of 19.6N with a Vickers microhardness indenter.

Results. The results from Vickers hardness were the percentage of delamination G1: 55%, G2: 50%, G3: 35%, G4: 0% and G5: 0%. SEM examination showed that the mean thickness of the reaction layer were G1 93.5±20.6㎛, G2 69.9±14.3㎛, G3 59.2±20.2㎛, G4 0.61±1.44㎛ G5 0±0㎛. The mean interfacial delamination crack lengths were G1 131±54.5㎛, G2 85.2± 51.3 ㎛, and G3 94.9±81.8㎛. One-way ANOVA showed that there was no statistically significant difference in interfacial crack length among G1, G2 and G3($p > 0.05$).

Conclusion. The investment reaction layer played important role at the interfacial toughness of body ceramic bonded to Lithia-based ceramic.

Key Words
Interfacial fracture toughness, Delamination, Bilayered ceramic, Reaction layer, Investment

The author acknowledges the generous advice received from Professor KJ Anusavice during the study. This research was supported by the Chonbuk National University Research Grant of 2004.

The fracture toughness of dental ceramics is determined mainly by the size and sharpness of flaws and by the resistance of cracks to propagation. Interfacial toughness is an important property that identifies the interfacial integrity of bonded materials. Common test methods for measuring fracture toughness of brittle materials include double cantilever beam, double torsion beam, single-edged notched beam (SENB), single-edged precracked beam (SEPB), chevron- notched beam (CNB), surface crack in flexure (SCF), indentation strength in bending (ISB), and indentation fracture (IF).[1] The test procedure for indentation fracture toughness is popular because of its simplicity, the benefit of a small sample size, and the potential for making repeat measurements on each specimen.[2] This technique has recently been used to assess the fracture toughness of various dental ceramics.[3-6]

Kelly et al. reported that crack initiation occurred at the core-veneer interface for approximately 70-78% of fractured all-ceramic (In-Ceram) fixed partial denture connectors, indicating that the interface is both a location of high tensile stress and an important source of structural flaws.[7] Interfacial bonding properties greatly affect the mechanical durability of laminated structures.[8] Indentation-induced delamination between veneer and core ceramics is possible using microhardness indentations to induce cracks. Delamination is most likely to occur by the growth of lateral cracks along the interface when the bonding integrity between core and veneer ceramic is not strong. A major distinguishing feature of a high quality bilayer interface is evidence of crack propagation across the interface rather than along the interface.

Vickers indentations made along a line parallel to specimen cross-sections demonstrate the capacity of the core ceramic layer to arrest radial and cone cracks extending from the adjacent veneer layer.[9] The interfaces between adjacent layers must be strongly bonded to prevent delamination. Cracks in the veneer layers can penetrate either along the interfacial region or into the adjacent core layers. This theory has been tested previously applied to a limited extent by crack propagation studies of ceramic/ceramic laminates.[10-11]

In this paper indentation fracture at interfaces between ceramic core and veneer materials is observed. The core and veneer materials were bonded together to form a well-defined, strongly bonded interface. However, when the reaction layer remains on the core surface, the interfacial bond may be unacceptable. Vickers indentations will be used to introduce controlled cracks in the veneer layer close to interface. In some situations where poor bonding has occurred, cracks may cause delamination along an interfacial path, or they may shield by a distributed damage zone in the reaction layer. In other configurations these cracks may propagate across the interface.

The purpose of this study was to determine the effect of the reaction layer on the interfacial fracture toughness of a core/veneer structure according to five different divesting procedures.

MATERIALS AND METHODS

A heat-pressed lithia-based ceramic (IPS Empress 2 core, lot # C11261 Ivoclar, Schaan, Liechtenstein) and a matching veneer (shade 210. lot # B05741) were selected for the study. Resin patterns, 20mm × 3 mm × 2 mm, were made from a polyvinyl siloxane impression. Thirty-five resin pattern bars were invested and burned out to produce IPS Empress 2 core bars according to the manufacturer's instructions. Rough divestment was carried out with a polishing jet medium (80㎛ glass beads, Garrenco, Henry Schein Inc., Indianapolis. USA) at 4 bars of pressure. For fine divestment, a pressure of 2 bars was applied. The sprue side of the core bars was ground with a diamond

disk and polished to a mean thickness of 2 mm by means of 240- to 600-grit SiC metallographic paper on a steel-backed metallographic polishing wheel. The bars were randomly divided into five groups of four each.

Five groups of four specimens each were prepared for the following surface divesting conditions:

Group 1: No dissolution or sandblasting of the interaction layer (to remove residual investment), rinse for 30s, and dry with oil-free air.

Group 2: Dissolve layer with 0.2% HF in an ultrasonic unit for 15 min (to remove residual investment), rinse for 30s, and dry with oil-free air.

Group 3: Dissolve layer with 0.2% HF in an ultrasonic unit for 30 min, rinse for 30s, and dry with oil-free air.

Group 4: Dissolve layer with 0.2% HF in an ultrasonic unit for 15 min, and rinse for 30s, dry with oil-free air, and sandblast the white reaction layer with a special jet medium (Al_2O_3, Type100) at 1 bar pressure for complete removal of investment for 60s, rinse for 30s, and dry with oil-free air.

Group 5: Dissolve layer with 0.2% HF in an ultrasonic unit 30 min, rinse for 30s, dry with oil-free air, sandblast the white reaction layer with a special jet medium (Al_2O_3, Type100) at 1 bar pressure for complete removal of investment for 60 s, rinse for 30 s, and dry with oil-free air.

Sandblasting was performed carefully with 100 ㎛ Al_2O_3 (Williams A Division of Ivoclar North America Inc. Amherst, New York) at 1 bar pressure until the white reaction layer was totally eliminated. All specimens were rinsed with tap water for 30s, and dried with oil-free air. Twenty core specimens were veneered with 1.8 mm of Empress 2 veneering ceramic according to the manufacturer's instructions. A silicone mold, approximately 4.5 mm × 3 mm × 20 mm, was used to form the bilayered core/veneer porcelain specimens prior to firing. Veneering porcelain was placed on the core specimen into the mold and condensed by the Vibra II handpiece (J.F. Jelenko & Co.) while excess moisture was blotted from the body porcelain with a paper tissue. The excess porcelain was then trimmed away with a straight-edged razor blade until the top surface of the porcelain was flush with the top surface of the mold. The veneered bars were fired with one wash firing and three body veneer firings according to the manufacturer's instruction. The specimens were made to a total thickness of 3.8 mm (2.0 mm core and 1.8 mm veneer). The bar surfaces were polished using 400- to 2000-grit SiC metallographic paper (Buehler Ltd., Lake Bluff, IL) and finished with 1㎛ polishing alumina (Mark V Laboratory, East Granby, CT, USA) until the specimens had a planar reflective surface. Each bar was polished to final dimensions of 20 mm × 2.8 mm × 3.8 mm. Each bar was ultrasonically cleaned in distilled water for 10 min after polishing. The resulting interface between the two ceramic layers was well-defined, with no visible voids.

The indentation technique is based on a series of cracks that form under heavy loading in a brittle material around a Vickers diamond indenter. A Vickers microhardness tester (Tukon Microhardness tester, Model MO, Page Wilson Corporation, Binghamton, NY 13905) was employed to induce cracks within the veneer close to interface. Each bar was indented at a load of 19.8 N in ambient air and received five indentations on the veneer surface close to the core/veneer interface bonding line. The indentations were placed at five widely separated locations 3 mm apart on each disk. Thus, 20 measurements were made for each group. The pyramidal indenter was oriented so that the two radial cracks were aligned parallel and perpen-

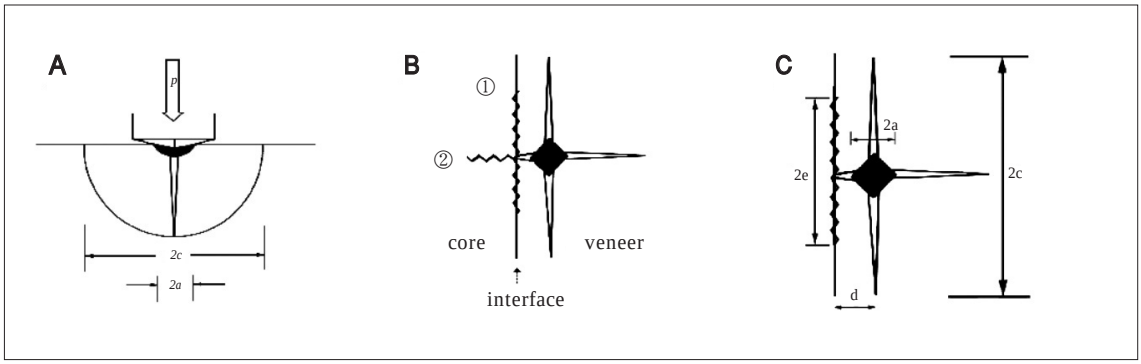

Fig. 1. Schematic diagram illustrating, A: Vickers indentation B: Crack penetrates core ceramics, and C: crack propagates along interface region, showing peak load, p, distance, d, delamination crack length, e, the characteristic dimensions of c and a of radial crack and the hardness impression, respectively.

dicular to the interface. Crack measurements were made immediately following removal of the indenter, usually after 30 s in air. The bars were then stored in distilled water at 37° C. Crack measurements were made also at 24 h, 72 h, and 128 h.

The crack patterns were considered acceptable under the following conditions: (1) all cracks emanated from the corner of the Vickers indentation; (2) no chipping occurred at the indent side; (3) no crack branching was present, (4) indentation did not contact the interface. The delamination crack length (2e) was obtained from measurements along the interfacial bonding line (Fig. 1). Readings were performed within 60 s after indentation using the indenter microscope at 200 X magnification.

Three core specimens from each group were randomly selected from the seven specimens and analyzed by SEM for evidence of residual reaction layer components resulting from the investment interaction. They were sectioned using a low-speed diamond saw, and polished using 2000 grit SiC paper. They were sonicated in ethyl alcohol for 30 min to eliminate surface debris. The cross section was secured to an aluminum block plate with mounting paste. They were dried at 37 ℃ in an oven overnight and coated with palladium prior to examination by scanning electron microscopy. The thickness of the reaction layer was measured for each group.

RESULTS

Vickers microhardness indentations were placed on the side of veneer layer close to the interface in core-veneer ceramic at a load of 19.8 N. When the reaction layer remained on the surface of bars in groups 1-3(Fig. 2), the indentation cracks either terminated at the interface or did not reach the interface when the indenter was placed far from the interface, i.e., when d >3 a(Fig. 3). When the indenter was placed directly on the interface, surface chipping and crushing was observed. The maximum amount of debonding was observed when the indentations were in the range of a < d < 3a. The interfacial crack lengths were independent of the distance, d, within this range. However, when the reaction layer was totally removed, as in groups G4 and G5 (Fig. 2), the cracks propagated across the interface (Fig. 3). The percentage of interfacial cracks and mean thickness of the reaction layer for each group are given in Table Ⅰ. SEM examination showed that the mean thickness of the reaction layer were G1 93.5±20.6㎛, G2 69.9±14.3 ㎛, G3 59.2±20.2㎛, G4 0.61±1.44㎛ G5 0±0㎛

m(Table Ⅰ & Fig. 3).

The mean interfacial crack length and standard deviation values are listed in Table Ⅱ. One-way ANOVA was performed to determine whether the differences existed between mean interfacial crack lengths were statistically significant, no significant differences were found among G1, G2 and G3 and as function of time for each group ($p > 0.05$).

Interfacial crack propagation occurred in Groups 1, 2, and 3, and crack propagation into the core occurred for groups 4 and 5 (Table Ⅱ & Fig. 3). The

Table Ⅰ. Percentage of interfacial cracks and the reaction layer thickness (㎛) for the five specimen groups

		G1	G2	G3	G4	G5
Number of Indentations		20	20	20	20	20
Percentage of Delaminations	Initial	55%	50%	35%	0%	0%
	24 h	60%	50%	35%	5%	0%
	72 h	65%	60%	35%	5%	0%
	128 h	70%	65%	40%	5%	0%
Thickness of reaction layer (㎛) (Mean±SD)		93.5±20.6	69.9±14.9	59.2±20.0	0.61±1.44	0±0

Table Ⅱ. Interfacial crack length (㎛)

	G1 Mean±SD	G2 Mean±SD	G3 Mean±SD	p value
Initial	131±54.5	85.2±51.3	94.9±81.8	0.215
24 h	140±55.5	99.7±55.0	97.8±81.6	0.263
72 h	140±55.0	105.3±55.3	101±83.8	0.346
128h	141±55.8	106±55.7	104±86.1	0.377

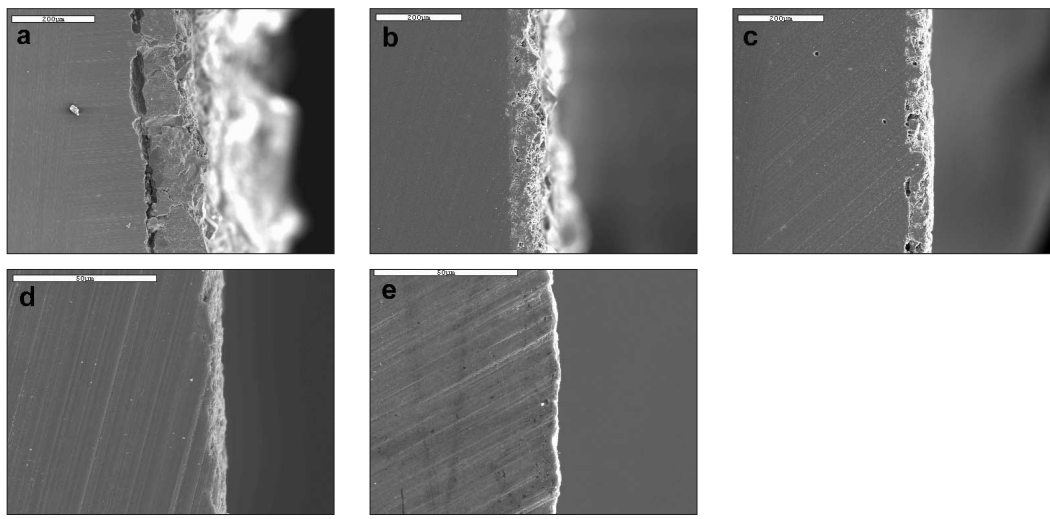

Fig. 2. SEM shows the reaction layers after 5 different divesting a: G1 b: G2 c: G3 d: G4 e: G5.

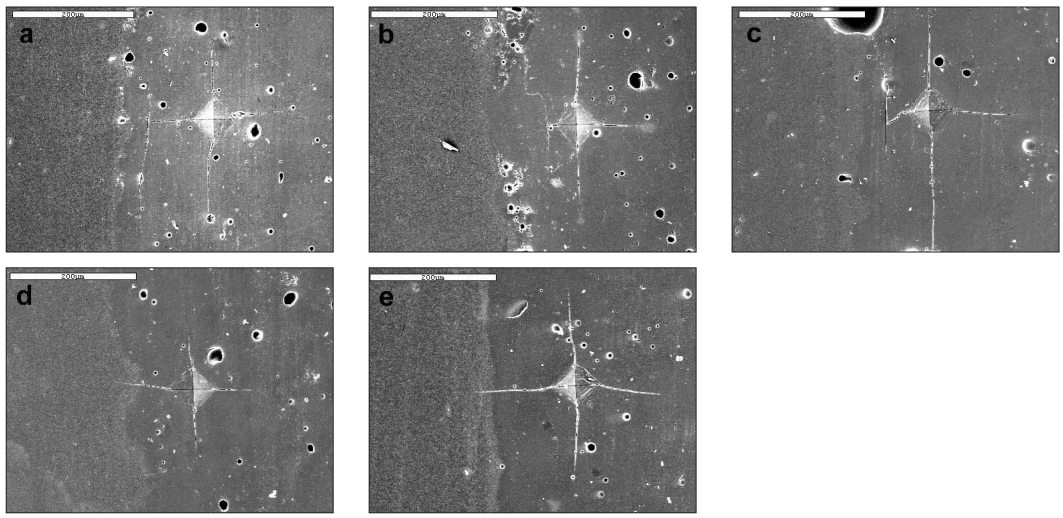

Fig. 3. SEM of core-veneer ceramics showing interfacial debonding G1-G3, and crack propagation into core for groups G4 and G5 produced by Vickers microindentation placed in veneer layer. a: G1 b: G2 c: G3 d: G4 e: G5.

reaction layers for all groups are shown in figure 2. Typical crack patterns for all groups are shown in figure 3.

DISCUSSION

To measure the radial (Palmqvist) surface crack lengths, the test surface must be polished until it is optically reflective before indentations are placed. The application of the Vickers indentation fracture toughness test to brittle materials, particularly glasses and ceramics, has become widespread because (1) it can be used on small samples of material not amendable to other fracture toughness tests; (2) specimen preparation is relatively simple requiring only the provision of a polished, reflective plane surface; (3) the Vickers diamond indenter used to produce the hardness indentations is a standard device used on a dedicated hardness tester or on a universal testing machine; (4) the crack lengths can be measured optically without undue difficulty in most cases; and (5) the test is rapid and cost effective.[12]

After heat pressing of lithia disilicate ceramic, a surface reaction layer is formed at the interface between the investment material and pressed ceramic. This white reaction layer is later removed using an acid solution and/or sandblasting, according to the manufacturer's instructions. Bilayered ceramic laminate structures have been proposed as a means of counteracting brittleness under tensile loading conditions by deflection of transverse cracks along an orthogonal interlayer path. Interfacial cracks of this kind are readily amenable to fracture mechanics analysis, by treating each member of the laminate as a continuum slab separated from its neighbors by weak interfaces. However, the interfaces between adjacent layers are strongly bonded and they inhibit delamination. Cracks in the veneer layer then penetrate the interfaces, become arrested in the adjacent core layer, or propagate across the interface.

Thompson reported that interfaces play an important role in the mechanical performance of biomaterial ceramic composites such as core-

veneer all-ceramic structures.[13] When the interface toughness exceeds the flexural stresses in the tensile surface at failure, a sharp crack will propagate and penetrate across the core-veneer interface, essentially behaving like a homogeneous material. Alternatively, when flexural stresses at failure exceed the interface toughness, the crack may deflect and extend along the interface between core and veneer.

Scanning electron microscopy is ideal for the analysis of divested surfaces because of its superior depth of field when compared with optical microscopy, and because of its ability to resolve features with dimensions less than 1 ㎛. SEM examinations showed that the mean thickness of the reaction layer for groups G1 to G5 were 93.5 ± 20.6 ㎛, 69.9 ± 14.9 ㎛, 59.2 ± 20.0 ㎛, 0.61 ± 1.44 ㎛, and 0 ± 0 ㎛ respectively. The reaction layer that remained after divestment was not completely removed by 0.2% HF acid solution regardless of time in groups 1-3. Sandblasting should be performed carefully until the white reaction layer was completely eliminated like group 5 for strong interfacial bonding between core and veneer. The investment reaction layer played important role the interfacial toughness of body ceramic bonded to Lithia-based ceramic.

Next experiment is in progress to finely calibrate the relationship between the interfacial toughness and the interfacial crack length.

CONCLUSIONS

Within the limitation of this study, the following conclusions were drawn: Etching with 0.2% HF alone did not eliminate reaction layer, but additional sandblasting was effective in removing the reaction layer. The presence of the reaction layer adversely affects the interfacial zone integrity but the thickness of the reaction layer has no effect on interfacial integrity.

REFERENCES

1. Fischer H, Marx R. Fracture toughness of dental ceramics: comparison of bending and indentation method. Dent Mater 2000;18:12-9.
2. Ponton CB, Rawlings RD. Vickers Indentation Fracture Toughness Test, Part 1: Review of literature and formulation of standised indentation toughness equations. Mat Sci Technol 1989;5:865-72.
3. Ponton CB, Rawlings RD. Vickers Indentation Fracture Toughness Test, Part 2: Application and critical evaluation of standardized indentation toughness equations. Mat Sci Technol 1989;5:961-76.
4. Anusavice KJ, Dehoff PH, Hojjatie B, Gray A. Influence of Tempering and Contraction Mismatch on Crack development in Ceramic Surface. J Dent Res 1989;68:1182-7.
5. Morena R, Lockwood PE, Fairhurst CW. Fracture toughness of commercial dental porcelains. Dent Mater 1986;2:58-62.
6. Seghi RR, Denry IR, Rosenstiel SF. Relative fracture toughness and hardness of new dental ceramics. J Prosthet Dent 1995;74:145-50.
7. Kelly JR, Tesk JA, Sorensen JA. Failure of All-ceramic Fixed Partial Dentures in vitro and in vivo: Analysis and Modeling. J Dent Res 1995;74:1253-8.
8. Wakabayashi N, Anusavice KJ. Crack Initiation Modes in Bilayered Alumina/ Porcelain Disks as a Function of Core/ Veneer Thickness Ratio and Supporting Substrate Stiffness. J Dent Res 2000;79: 1398-404.
9. Wuttiphan S, Lawn BR, Padture NP. Crack Suppression in strongly Bonded Homogeneous/ Heterogeneous Laminates: A Study on Glass/Glass-Ceramic Bilayers. J Am Ceram Soc 1996;79:634-40.
10. Rossington C, Evans AG, Marshall DB, Khuri-Yakub BT. Measurements of adherence of residually stressed thin films by indentation, II: Experiments with ZnO/Si. J Appl Phys 1984;56:15.
11. Marshall DB, Evans AG. Measurement of adherence of residually stressed thin films by indentation, I: Mechanics of interface delamination. J Appl Phys 1984;56:15.
12. Anstis GR, Chantikul P, Lawn BR, Marshall DB. A Critical Evaluation of indentation Technique for Measuring Fracture Toughness: I, Direct Crack measurements. J Am Ceram Soc 1981;64:533-8.
13. Thomson GA. Influence of relative layer height and testing method on the failure mode and origin in a bilayered dental ceramic composite. Dent Mater 2000;16:235-43.

Reprint request to:
JU-MI PARK, D.D.S.,M.S.D.,PH.D.
DEPARTMENT OF PROSTHODONTICS, SCHOOL OF DENTISTRY,
CHONBUK NATIONAL UNIVERSITY
664-14 DUKJIN DONG, DUKJIN-GU, JEONJU, 561-756, KOREA
jmpark@chonbuk.ac.kr