Effect of Resin Luting Film Thickness on Fracture Resistance of a Ceramic Cemented to Dentin

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Purpose: **The aim of this study was to evaluate the fracture resistance of ceramic plates cemented to dentin as a function of the resin cement film thickness.**

Materials and Methods: **Ceramic plates (1 and 2 mm thicknesses) were cemented to bovine dentin using resin composite cement. The film thicknesses used were approximately 100, 200, and 300 μm. Noncemented ceramic plates were used as control. Fracture loads (N) were obtained by compressing a steel indenter in the center of the ceramic plates. ANOVA and Tukey tests ($\alpha = 0.05$) were used for each ceramic thickness to compare fracture loads among resin cement films used.**

Results: **Mean fracture load (N) for 1-mm ceramic plates were: control—26 (7); 100 μm—743 (150); 200 μm—865 (105); 300 μm—982 (226). Test groups were significantly different from the control group; there was a statistical difference in fracture load between groups with 100 and 300 μm film thicknesses ($p < 0.01$). Mean fracture load for 2-mm ceramic plates were: control—214 (111); 100 μm—1096 (341); 200 μm—1067 (226); 300 μm—1351 (269). Tested groups were also significantly different from the control group ($p < 0.01$). No statistical difference was shown among different film thicknesses.**

Conclusions: **Unluted specimens presented significantly lower fracture resistance than luted specimens. Higher cement film thickness resulted in increased fracture resistance for the 1-mm ceramic plates. Film thickness did not influence the fracture resistance of 2-mm porcelain plates.**

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INDEX WORDS: all-ceramic crown, dentin-bonded crown, fracture strength, resin-based luting systems, ceramics

METAL-CERAMIC CROWNS HAVE BEEN used for over 40 years to provide strong and functional restorations. Recently, the trend has been to eliminate this metal substructure, because these restorations present drawbacks, such as poor esthetics, as they may appear gray, opaque,

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or chalky.[1] Some patients may experience soft tissue allergic reaction to metal. Exposure of a visible dark line may also result from subsequent gingival recession.[2]

The principal and inherent mechanical characteristics of ceramic materials are their hardness, brittleness, high wear resistance, and low flexural and tensile strength.[3] Ceramic materials fracture at a fraction of their theoretical strength, due to the presence of microscopic flaws that act as local stress concentration sites.[1] To overcome this problem, metal-free ceramic restorations should be bonded to the tooth structure with strong cements. It has been shown that inlays cemented with adhesive materials have superior fracture resistance compared with those cemented with conventional cements.[1,4,5]

The effect of resin cement thickness on the fracture resistance of all-ceramic restorations is not well established. The influence of film thickness of resin luting agents on the joint bend strength of the ceramic/dentin interface has been measured,[6] showing that bond strength values were significantly lower for the 20-μm film than for thicker films. Scherrer et al[7] reported the effect of cement film thickness on the fracture resistance of ceramic plates loaded in compression using a spherical indenter. They found that the fracture resistance of glass ceramic cemented with zinc phosphate cement was not dependent on film thickness. When the resin cement was used, gradual decrease of the fracture strength, which became statistically significant at a cement thickness of 300 μm or more, was observed. The different film thicknesses were obtained by changing the cement viscosity as a function of time and the amount of load applied during cementation. According to another study,[8] which evaluated the crack propensity of laminated veneers subjected to thermocycling (5°C to 50°C for 1000 cycles), a thick ceramic veneer combined with minimal thickness of luting composite was shown to provide restorations with a favorable configuration with regard to crack propensity, namely, a ceramic and luting composite thickness ratio above 3.0. For instance, in this study, the facial ceramic thickness (C) and luting composite thickness (LC) ratio (R = C/LC) was about 3.9 ± 0.19 (R = 900 μm/230 μm) for noncracked specimens and 2.6 ± 0.35 (R = 700 μm/270 μm) for cracked specimens. The facial ratio (R) was above 3.0 for 14 of the 16 noncracked specimens, and below 3.0 for 9 of the 11 cracked specimens.[8]

The purpose of this study was to address the question of whether cement film thickness significantly influences the compressive fracture resistance of a ceramic under different standardized cement thicknesses. The null hypothesis tested was that resin cement film thicknesses have no influence on the compressive fracture resistance of ceramic plates cemented to dentin.

Materials and Methods

Experimental Design

The factors under study were four resin luting agent thicknesses (no cementation, 100, 200, and 300 μm) and two ceramic thicknesses (1 and 2 mm). The association between luting agent and ceramic thicknesses resulted in eight groups (4×2). The experimental sample consisted of 80 specimens (n = 10), made in random sequence. The response variable was ceramic fracture resistance evaluated by means of compressive strength test.

Materials

The materials used for this study are listed in Table 1.

Preparation of Teeth

Eighty bovine mandibular incisors stored at 4°C in a solution of 0.5% chloramine T were used. Only teeth that were sound and free from defects and cracks on visual examination were included. The teeth were embedded in epoxy resin (RD-6921, Redelease, SP, Brazil), and a flat superficial dentin surface was exposed by wet grinding with 320 and 600-grit silicon carbide abrasive papers.

Table 1. Materials Used

Materials	*Manufacturer*	*Batch Code*
Duceram Plus	Degussa	0090/22
Rely X ARC	3M ESPE	3415A3
Single Bond	3M ESPE	1105
35% Phosphoric acid	3M ESPE	7523
Silane agent	3M ESPE	0086
10% Hydrofluoric acid	Dentsply	68758

Preparation of Ceramic

Ceramic blocks (Duceram-Plus, Degussa, Rosbach, Germany) were fabricated by condensing the material in a glass mold. The condensed blocks were fired in a ceramic oven according to the manufacturer's instructions. They were cut into rectangular slices (3.0 × 6.0 mm) in two thicknesses, 1 and 2 mm, using a low-speed diamond saw (South Bay Technology Inc., model 650, San Clemente, CA) under water-cooling. The slice thicknesses were measured with a digital caliper (Mitutoyo, Japan) in three locations to ensure uniform rectangular ceramic plates. Specimens were kept in distilled water at 37°C until they were cemented to dentin. Before cementation, they were etched with 10% hydrofluoric acid for 60 seconds (Dentsply, Petrópolis, RJ, Brazil), washed for 60 seconds, dried, silanized (Ceramic Primer, 3M ESPE, St. Paul, MN) for 60 seconds, and lightly air-thinned.

Tested Groups

The groups tested in this study are listed in Table 2.

Specimen Design and Cementation Procedure

The complex geometry of a full molar crown makes it extremely difficult to determine the quantitative fracture strength under occlusal loading; hence, the current study used a test design with simple specimen geometry: a point load onto a uniformly supported rectangular ceramic plate[7] luted to dentin.

Two metal strips (positioned at opposite sides of each ceramic plate) of the desired gap width serving as spacer to control the cement film thickness[9] and luted area (3.0 × 5.0 mm) separated the dentin and the ceramic plates from each other. The three cement films selected for this study for each ceramic thickness were 100, 200, and 300 μm. Before cementation, the dentin was etched for 15 seconds with 35% phosphoric acid (3M ESPE), washed for 30 seconds and dried with absorbent papers. Next, the Single-Bond (3M ESPE) adhesive system was applied to the surface and polymerized for 20 seconds (Optilux Demetron, VLC 403, delivering 500 mW/cm,^{2} Danbury, CT). The gaps between the dentin and ceramic plates were filled with the resin luting cement (Rely X ARC, 3M ESPE) mixed according to the manufacturer's instructions. A load of 1 kg was applied to the ceramic plates during photopolymerization (40 seconds for each free surface). Each specimen was stored in deionized water at 37°C for 24 hours prior to testing, to allow any immediate post-cure polymerization of the luting agent to occur.

Table 2. Studied Groups

Groups	*Ceramic Plate Thickness (mm)*	*Cement Thickness*
A	1	No cementation procedure
B	1	100 μm
C	1	200 μm
D	1	300 μm
E	2	No cementation procedure
F	2	100 μm
G	2	200 μm
H	2	300 μm

Compressive Test

The specimens were subjected to compressive loading at a crosshead speed of 0.5 mm/min in a Universal Testing Machine (EMIC DL 500, São José dos Pinhais, PR, Brazil). Compressive force was applied by means of a 2-mm diameter steel indenter placed in the center of the ceramic plate (previously marked). The testing machine crosshead was stopped when the first discontinuity of the chart recording appeared, as a result of an early crack or catastrophic failure. The compressive force required to cause fracture of the ceramic plates was recorded in Newtons (N).

Scanning Electron Microscopic Examination

Representative samples were selected for microscopic evaluation of the luting agent thickness. Luted specimens were transversally cut using a low-speed diamond saw under water-cooling (South Bay Technology Inc.). Cementation interfaces were wet polished with 600, 1000, and 1200 grit silicon carbide abrasive papers and 1, 0.3, and 0.05 μm finishing oxide aluminum pastes (Arotec S/A Ind e Com., São Paulo, Brazil) for 30 seconds each. The surfaces were carefully washed with water and ultrasonically cleaned for 1 minute after each polishing step. Each specimen was demineralized with 6 M hydrochloric acid for 20 seconds and deproteinized with 2% NaOCl for 1 minute. They were left to dry for 24 hours at 37°C and allowed to air-dry completely in a desiccator. Specimens were mounted on aluminum stubs, sputter-coated with gold (MED 010, Balzers, Liechtenstein), and observed under a scanning electron microscope (LEO 435 VP, Cambridge, England) at 350× magnification.

Statistical Analysis

One-way ANOVA and Tukey tests at the 95% confidence interval were used for each ceramic thickness to compare fracture loads among different resin films.

Table 3. Mean Fracture Load (N), Standard Deviation, and Statistical Analysis for 1-mm Ceramic Plate Groups

Groups	*MFL (s.d.)*	*Tukey*		
A (1 mm; control)	26 (7)	α		
B (1 mm; 100 μm)	743 (150)		β	
C (1 mm; 200 μm)	865 (105)		β	γ
D (1 mm; 300 μm)	982 (226)			γ

MFL = mean fracture load; n = 10; $\alpha = 0.05$; different Greek letters indicate statistical differences among groups.

Results

Mean fracture loads together with standard deviations for specimens cemented with each of the ceramic thicknesses and resin cement films are shown in Tables 3 and 4. The ANOVA test was carried out on the fracture load data for each ceramic thickness (1 mm, 2 mm). Examination of the homogeneity of variance indicated that data met the assumptions of this test. The multiple comparisons were carried out using the Tukey test. For 1-mm ceramic plates, the data for luted specimens were significantly different from the control group. A significant increase in fracture resistance between 100 and 300 μm resin cement film groups was shown ($p < 0.01$). For 2-mm ceramic plates, the data for luted specimens were significantly different from the control group ($p < 0.01$). No difference was shown among groups with different resin cement film thickness.

The scanning electron microscopy observations identified the cement film thickness of specimens fabricated with 100 μm (Fig 1—103.52 μm), 200 μm (Fig 2—205.29 μm), and 300 μm (Fig 3—318.46 μm) spacers.

Table 4. Mean Fracture Load (N), Standard Deviation, and Statistical Analysis for 2-mm Ceramic Plate Groups

Groups	*MFL (s.d.)*	*Tukey*	
E (2 mm; control)	214 (111)	α	
F (2 mm; 100 μm)	1096 (341)		β
G (2 mm; 200 μm)	1067 (226)		β
H (2 mm; 300 μm)	1351 (269)		β

MFL = mean fracture load; n = 10; $\alpha = 0.05$; different Greek letters indicate statistical differences among groups.

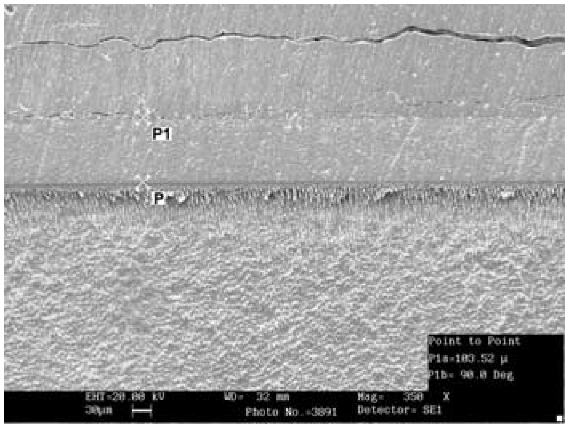

Figure 1. Representative sample fabricated with 100-μm spacer (obtained cement thickness P-P1 = 103.52 μm) (350×).

Discussion

The geometry of a tooth crown makes quantitative determination of the effect of resin luting film thickness on the fracture resistance of ceramic under occlusal loading extremely difficult. Hence, the current study used a specimen design with a simple geometric configuration in which a point was loaded on identical and uniformly supported rectangular ceramic plates[7] luted to dentin with different resin cement film thicknesses. Microscopic observations of luting film thickness in this study showed minimal variations (Figs 1–3),

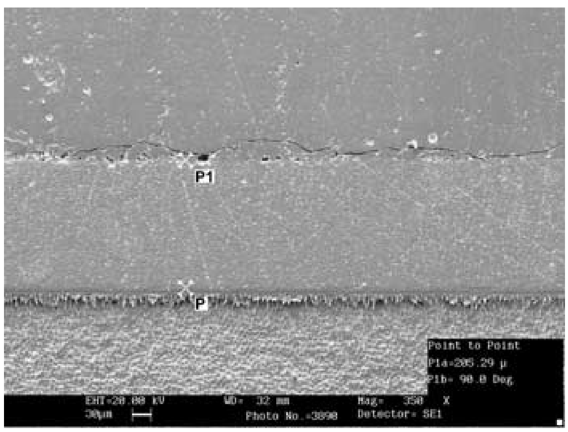

Figure 2. Representative sample fabricated with 200-μm spacer (obtained cement thickness P-P1 = 205.29 μm) (350×).

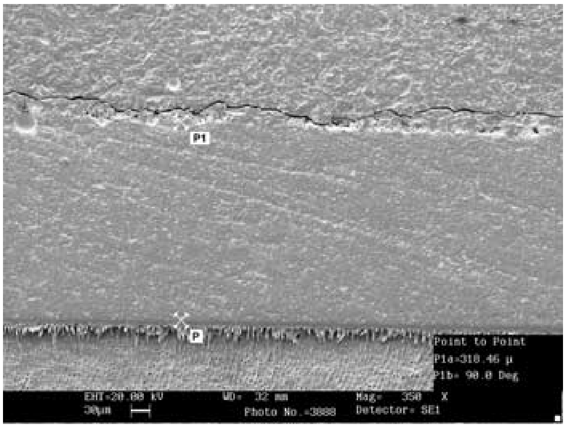

Figure 3. Representative sample fabricated with 300-μm spacer (obtained cement thickness P-P1 = 318.46 μm) (350×).

indicating that a reliable method for controlling resin cement thickness was used.

For both ceramic plate thicknesses, the luted specimens presented significantly higher fracture resistance than the unluted specimens. These results were expected, since ceramic materials are brittle and possess low flexural and tensile strengths,[3] and specimens were not supported by a cement layer. In addition, cementing ceramics with resin luting agents provides a means of stress transfer from ceramic to resin cement, from resin cement to bonding agent, from bonding agent to hybrid layer, and from hybrid layer to dentin. According to Burke and Watts[10] the components of this system represent the materials of choice for luting ceramic crowns.

The present study also demonstrated that for 1-mm ceramic plates, the increase in resin cement film gradually resulted in a higher ceramic fracture resistance. Conversely, different resin cement films did not influence fracture resistance of 2-mm ceramic plates. In view of these results, the null hypothesis was rejected. According to Thompson and Rekow,[11] when the ceramic is thick, bulk properties dominate in its upper portion. Here glass cone cracking is observed. This behavior is independent of the substrate supporting the ceramic and is responsible for chipping and surface cracks in inlays and onlays as well as for veneering ceramics. When the ceramic thickness falls below about 1 mm, flexural radial cracking becomes predominant. In this case, the stiffness of the substrate, such as luting cements and tooth structure, plays a role in causing failure.

In Scherrer et al's investigation,[7] a relatively small (<10%) but statistically significant difference in strength was found only between the two extremes studied: film thickness groups of 26 and 297 μm (ceramic dimension: 12.5 × 12.5 × 2 mm). A slight downward trend of the fracture load with increasing resin cement thickness was observed. In the present study, for 2-mm thickness ceramic plates, no statistical differences were found among different resin cement film thicknesses. Nevertheless, it is difficult to make direct association between the two studies, as they used different specimen dimensions, types of ceramic, and resin cement systems. These are factors that can affect ceramic fracture resistance behavior.[12] In another study that evaluated the effect of resin cement on 2-mm ceramic crown fracture resistances, the luting material thickness of fractured specimens was observed.[10] The mean film thickness of the best performing material tested was similar to the group that did not perform as well. It was concluded that the film thickness did not influence the overall result. On the other hand, according to Kim et al,[13] increased cement thickness can have a large effect on reducing flexural failure load. In that study, the load to failure of silicon bonded to glass with variation in the thickness of the bonding epoxy layer indicated that by increasing this layer from 20 to 200 μm, there was a 50% reduction in strength. This system is analogous to a structural ceramic crown on dentin with variation in cement thickness.

Molin et al[6] evaluated the influence of film thicknesses of resin composite luting agents on the joint bend strength of two ceramic/resin interfaces. When the bond strength for the different film thicknesses within each ceramic-cement combination was analyzed, the values were significantly lower for the 20-μm film thickness than for 50, 100, and 200 μm. The authors state that the mixing procedure for a dual-cure luting material incorporates air into the bulk of the material. The incorporation of porosity in composites has been shown to reduce shrinkage stress as a consequence of increased free area inside the bulk.[14] Therefore, this porosity could be more prominent in thick layers distributing this stress more uniformly.[6] In addition, the contraction stress generated when luting ceramic inlays with composites might induce forces at the ceramic-resin interface, thereby influencing strength and longevity.[15] Reaching a critical magnitude, the setting stress might induce premature debonding of certain areas in the adhesive joint. This polymerization stress might be more significant in thin bonded resin layers due to unfavorable geometry.[16] The presence of defects at the bonding interface might be a contributory factor to the present findings, where a significant upward trend of the fracture load with increasing resin cement thickness was noted for 1-mm ceramic plates.

According to the manufacturer, the film thickness of the cement used in this study is 13 μm. This measurement was made by placing mixed cement between two plates and applying a load on top of the plate to determine how thin the cement layer would get. Clinically it is difficult to obtain such standardized cement film thicknesses. For resin cements combined with an adhesive system, the contribution of the adhesive layer film thickness must be taken into consideration. This is of particular concern with any adhesive system that is not self-curable and requires the adhesive layer to be light-cured prior to seating the indirect restoration.[17] The film thickness of a luting agent is also a function of the viscosity of the cement mixture. High-viscosity luting composites may adversely affect the optimal seating of an indirect restoration. Type of filler particles (macrofiller or microfiller), composition of resin matrix, and degree of polymerization define viscosity.[18] Moreover, the application of the die spacers during laboratory procedures may influence the luting agent film thickness. The omission of a die spacer may affect the proper seating of the restoration, and an excessive layer can also generate an enlarged luting space.[8] Finally, the luting agent thickness can be related to the material used for indirect restoration. For instance, a significant volumetric shrinkage occurs inherent to the firing process of conventional powder-liquid ceramics that varies from 15% to 40% depending on the material used.[19]

Conclusions

From the results of this study the following conclusions can be drawn:

1. The unluted specimens presented a significantly lower fracture resistance than the luted specimens.
2. Higher resin cement film thickness resulted in increased fracture resistance for the 1-mm ceramic plates.
3. The resin cement film thickness did not influence the fracture resistance of 2-mm ceramic plates.

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