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Microtensile Bond Strength of Glass Ionomer Cements to Artificially Created Carious Dentin

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Clinical Relevance

The mean microtensile bond strengths of glass ionomer cements to carious dentin were significantly lower than to sound dentin.

SUMMARY

In this laboratory study, the microtensile bond strengths of a conventional glass ionomer cement (GIC) and a resin modified glass ionomer cement (CRMGIC) to artificially created carious dentin and sound dentin were compared, and the ultrastructural morphology of the fractured interface was examined with a low-vacuum scanning electron microscope (SEM). The specimens were divided into 4 groups: 1) a conventional GIC (Ketac-Fil Plus Aplicap) placed on sound dentin; 2) a conventional GIC placed on artificially created carious dentin; 3) an RMGIC (Photac-Fil Aplicap) placed on sound dentin and 4) an RMGIC placed on artificially created carious dentin. Artificial carious lesions were created using a chemical demineralizing solution of 0.1 M/L lactic acid and 0.2% carbopol. GIC buildups were made on the dentin surfaces according to the manufacturer's directions. After storage in distilled water at 37°C for 24 hours, the teeth were sectioned vertically into 1 x 1 x 8-mm beams for the microtensile bond strength test. The microtensile bond strength of each specimen was measured, and failure mode was determined using an optical microscope (40x). The fractured surfaces were further examined with SEM. Two-way analysis of variance showed that the mean microtensile bond strengths of a GIC and an RMGIC to carious dentin were significantly lower than those to sound dentin, and the mean microtensile bond strengths of Photac-Fil to both sound and carious dentin were significantly higher than those of Ketac-Fil Plus. Chi-square tests indicated that there was a significant difference in failure mode between the sound dentin and carious dentin groups. In sound dentin groups, cohesive failure in GIC was pre-

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dominant; whereas, mixed failure was predominant in carious dentin groups. SEM examination showed that the specimens determined to be cohesive failures under light microscopy in the Photac-Fil/Sound Dentin group were actually mixed failures under high magnification of SEM.

INTRODUCTION

Since glass ionomer cement (GIC) was developed by Wilson and Kent in the early 1970s, it has been widely used in clinical dentistry. Its main advantages are direct chemical bonding to tooth substrate, long-term fluoride ion release and uptake, good biocompatibility to pulpal tissue and similar coefficients of thermal expansion with tooth structure. However, their inferior mechanical properties limit their use as a restorative material to non-stress-bearing areas.

In minimal intervention dentistry, some authors recommend excavating only the outer layer of highly infected, denatured, caries-infected dentin and preserving the inner layer of intact, bacteria-free, remineralizable, caries-affected dentin when a cavity is prepared for restoration.[1-6] In atraumatic restorative treatment (ART), complete removal of decay is not always possible, because only hand instruments are used for excavation of the carious lesion.[7] Incomplete caries excavation sometimes occurs in conventional cavity preparations. A significant number of restorative failures, reported as recurrent caries, are probably the result of residual caries.[8] GICs have often been used as the restorative material of choice in these situations. The clinical success rate of the ART approach is fairly high in initial, short-term studies.[9-10] However, no long-term studies are available yet, and few studies have been done on the bond strength and microscopic characteristics of the bonding interface between GICs and carious dentin.

This laboratory study examined the microtensile bond strength of a conventional GIC and an RMGIC to artificially created carious dentin and examined the ultra-structural morphology of the fractured interface with a scanning electron microscope (SEM).

METHODS AND MATERIALS

This study involved 4 groups.

Group 1. Ketac-Fil Plus Aplicap placed on sound dentin.

Group 2. Ketac-Fil Plus Aplicap placed on artificially created carious dentin.

Group 3. Photac-Fil Aplicap placed on sound dentin.

Group 4. Photac-Fil Aplicap placed on artificially created carious dentin.

Each group contained 3 teeth, with 9 specimens being prepared from each tooth. Each group had 27 specimens.

Materials

For this study, Ketac-Fil Plus Aplicap (Ketac-Fil, ESPE, Seefeld, Germany) and Photac-Fil Aplicap (Photac-Fil, ESPE) were used as representative materials of conventional GIC and RMGIC. Ketac Conditioner (ESPE), a 25% polyacrylic acid, was used as a conditioner.

Tooth Preparation

Twelve recently extracted caries and crack-free human molars were treated with 10% buffered formalin solution after extraction for up to 2 weeks, they were then stored in deionized water. The tooth surface was cleaned with periodontal curettes and stored in deionized water at 3-4°C until use. In order to expose a flat occlusal dentin bonding surface, the external enamel of each specimen was ground with a 180 grit silicon carbide paper mounted on a water-cooled wheel. The occlusal tooth surfaces were then examined with a microscope to confirm that no enamel structure remained. The teeth were progressively polished with moist silicon carbide sandpapers in grits of 240, 320, 400 and 600, passing 10 times for each grit. The grinding was standardized by rotating each tooth 90° after each grit. The polished surfaces were cleaned in an ultrasonic cleaner for 5 minutes and rinsed with a 3-way-syringe. Each tooth was mounted on 4 x 4 cm Plexiglass using green modeling compound. A jig was used to create parallelism between the occlusal surface and the Plexiglass. Two perpendicular lines were drawn on the Plexiglass to guide mounting the tooth specimens on an Isomet saw (Isomet, Buehler, Lake Bluff, IL, USA).

Creation of Artificial Caries

Six teeth were randomly assigned to the carious dentin groups. The whole tooth surface was coated twice with acid-resistant varnish, except for the occlusal dentin surface window. Artificial carious lesions were created using the method previously described by Toro-Arrivillage.[11] The teeth were immersed in 15 ml of a chemical demineralizing solution of 0.1 M/L lactic acid and 0.2% carbopol, which had been saturated with hydroxyapatite and adjusted to pH 5, at 37°C for 96 hours. The specimens were then carefully washed with tap water to remove all traces of the solution and stored in deionized water at 4°C until bonding.

Bonding Specimen Preparation

The tooth specimens were stored in 37°C deionized water for 24 hours before bonding. Each specimen was washed with deionized water and dried with canned-compressed air. The specimens were then conditioned with a Ketac Conditioner for 20 seconds and rinsed with deionized water for 10 seconds using a 3-way-syringe. The remaining water was blot-dried with gauze to prevent desiccation. The materials were mixed with a ProMix triturator (Dentsply Caulk, Milford, DE, USA), with a setting of 10 seconds on fast for Ketac-Fil and 15 seconds on fast for Photac-Fil and applied to the tooth surface until the whole dentin surface was covered. Additional increments of GICs were placed to form a 5-mm high core. For conventional GIC, the surface was coated with unfilled resin, Ketac Glaze (ESPE) and cured using an Elipar Highlight (ESPE) at 800-900 mW/cm^2 verified with a Model 100 Curing Radiometer (Demetron Research Corp, Danbury, CT, USA) to prevent desiccation, and the specimen was allowed to set for 10 minutes. For RMGIC, each 1-2 mm increment was cured for 40 seconds using the Elipar Highlight. After setting, all specimens were stored in 37°C deionized water for 48 hours before sectioning.

Each specimen was sectioned vertically with an Isomet saw through the GIC buildups and the dentin at 1-mm increments to produce a series of 1-mm thick slabs. Each slab was vertically sectioned into 1 mm x 1 mm x 8 mm beams.[12] The dentin base of each beam was cut with a thin diamond bur using a high-speed handpiece. Each beam was stored in 37°C deionized water until testing. Using this technique, each tooth yielded 9 beams for the bonding test.

Microtensile Bond Strength Test and Failure Mode Examination

The external bonding interface of each beam was coated with Vaseline to prevent cyanoacrylate (Zapit; DVA, Corona, CA, USA) intervention on the bonding area. Then, each beam was fixed to a modified Bencor Multi T testing assembly (Danville Engineering, San Ramon, CA, USA) using cyanoacrylate. A tensile force was given to the beam using a 1123 MTS Renew Testing Machine (Northfield, IL, USA) at a crosshead speed of 1-mm per minute until it failed.[13] The fractured surface of the specimen was examined with an optical microscope (40x) to determine the mode of failure, and the exact dimension of each beam was measured individually using a Measurescope UM-2 (Nikon, Tokyo, Japan).

The failure mode was classified as either adhesive between dentin and the GIC, cohesive within the GIC or mixed (a combination of adhesive failure along the dentin surface and cohesive failure in the GIC or dentin). Upon finishing this procedure, the specimens were kept at 100% humidity until SEM examination.

SEM Observation

The dentin side of the fractured beams from each group was examined using a JEOL JSM-5310LV Scanning Electron Microscope (JEOL Ltd, Akishima, Japan) without any specimen preparation.

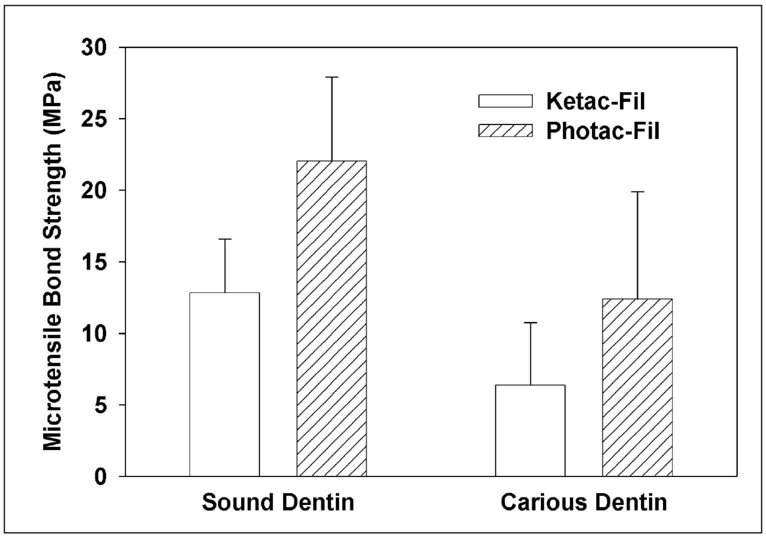

Figure 1. *The mean microtensile bond strengths of Ketac-Fil and Photac-Fil to sound and carious dentin. This graph shows the significant difference between the Ketac-Fil and Photac-Fil groups and between the carious dentin and sound dentin groups.*

Table 1: *Mean Microtensile Bond Strengths (MPa) ± SD*

Substrate \ Material	Ketac-Fil	Photac-Fil
Sound Dentin	12.81 ± 3.77	22.02 ± 5.90
Carious Dentin	6.36 ± 4.38	12.40 ± 7.48

Two-way ANOVA showed significant differences in microtensile bond strengths between sound and carious dentin for both Ketac-Fil and Photac-Fil (p<0.05).

Statistical Analysis

Bond strength data obtained from the 4 groups were analyzed using a 2-way analysis of variance (ANOVA) to find differences between the materials and the presence of artificial caries at a 95% confidence level. To learn differences in failure mode among the 4 groups, Chi-square tests were performed at the 0.05 probability level.

RESULTS

The mean microtensile bond strengths of a conventional GIC (Ketac-Fil) and an RMGIC (Photac-Fil) to sound dentin and carious dentin are shown in Figure 1 and Table 1. Two-way ANOVA showed that there were significant differences in microtensile bond strengths between sound and carious dentin for both Ketac-Fil and Photac-Fil ($p<0.05$). The mean microtensile bond strengths of a GIC and an RMGIC to carious dentin were significantly lower than those to sound dentin. The mean microtensile bond strengths of Photac-Fil to both sound and carious dentin were significantly higher than those of Ketac-Fil groups.

The specimen numbers of the different failure modes for each group are shown in Table 2. Chi-square tests indicated that there was a significant difference in failure mode between the sound and carious groups ($p<0.05$). In sound groups, cohesive failure in GICs was the predominant mode of failure; whereas, mixed failure was the predominant failure mode in carious groups. There was no significant difference in failure mode between Ketac-Fil and Photac-Fil for either the sound or carious groups ($p<0.05$).

DISCUSSION

Comparison of Bond Strengths

Not many studies have investigated the bond strength of GICs to carious dentin, and the study results have not been consistent. Some have shown that the mean bond strength of glass ionomer cement to carious dentin is similar to normal dentin.[14-15] Others have indicated that the bond strength of an RMGIC (Fuji II LC) to carious dentin was significantly lower than that to sound dentin.[16] The results of this study support the latter concept. Considering the chemical bonding mechanism between the carboxyl groups of the polyalkenoic acid and calcium ion of hydroxyapatite,[17] the lower mean bond strengths of a conventional GIC (Ketac-Fil) and an RMGIC (Photac-Fil) to carious dentin are reasonable, because loss of calcium ions through demineralization in the carious dentin groups gives less opportunity for bonding between calcium ions and the carboxyl groups.

Because of better mechanical properties and the formation of a hybrid-like layer, most studies have shown that the mean bond strength of RMGICs to sound dentin is significantly higher than that of conventional GICs. In this study, the mean bond strength of an RMGIC (Photac-Fil) to sound dentin is significantly higher than that of a conventional GIC (Ketac-Fil). This result is consistent with many previous studies.[18-19]

In this study, the mean microtensile bond strength of an RMGIC (Photac-Fil) to carious dentin is much higher than that of a conventional GIC (Ketac-Fil). This finding is consistent with the study by Palma-Dibb and others,[15] although the actual values from this study appear to be higher.

Low-Vacuum SEM Observations

Figure 2 shows the dentin-side of the interface in a cohesively failed specimen of the Ketac-Fil/Sound Dentin group. Because the material is covering the entire surface of dentin, no dentin surface can be seen. Numerous air bubbles trapped in the material also are seen. Many cracks evident within the material are attributed to dehydration during the SEM procedure. Figure 3 shows a typical mixed failure mode in the Ketac-Fil/Sound Dentin group.

Table 2: *The Specimen Numbers of Different Failure Modes for Each Group*

Group \ Failure Mode	Cohesive	Mixed	Adhesive
Ketac-Fil + Sound Dentin	20	7	0
Ketac-Fil + Carious Dentin	4	21	2
Photac-Fil + Sound Dentin	24	2	0
Photac-Fil + Carious Dentin	8	17	0

Chi-square tests indicated significant differences in failure mode between sound groups and carious groups (p<0.05).

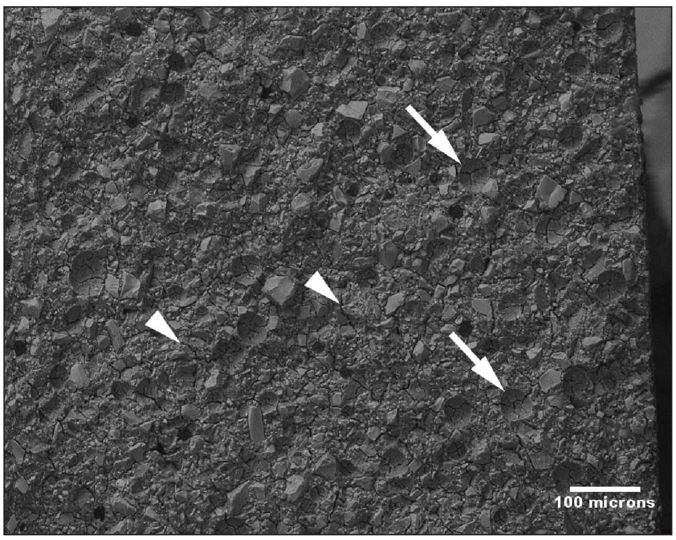

Figure 2. *Cohesively failed specimen in the Ketac-Fil/Sound dentin group. The white arrows show the air bubbles and the white arrowheads show the crack lines. These crack lines were created due to dehydration during SEM observation.*

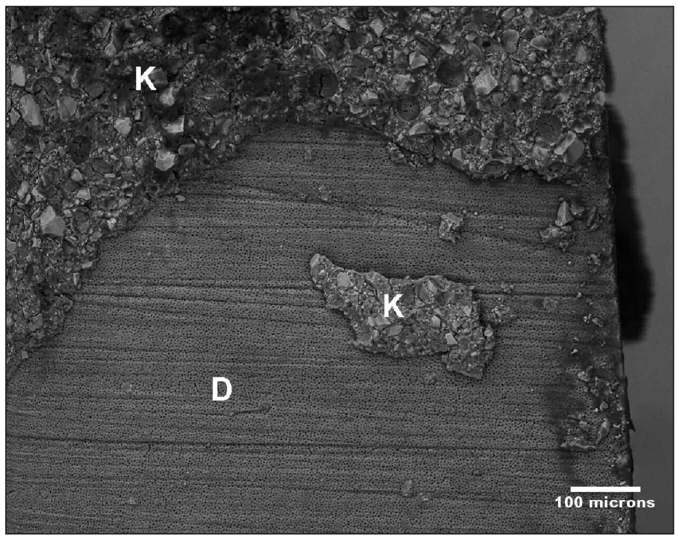

Figure 3. *Mixed failure specimen in the Ketac-Fil/Sound dentin group. K: Cohesively failed region within Ketac-Fil. D: Adhesively failed region between Ketac-Fil and dentin. Dentin surface was exposed.*

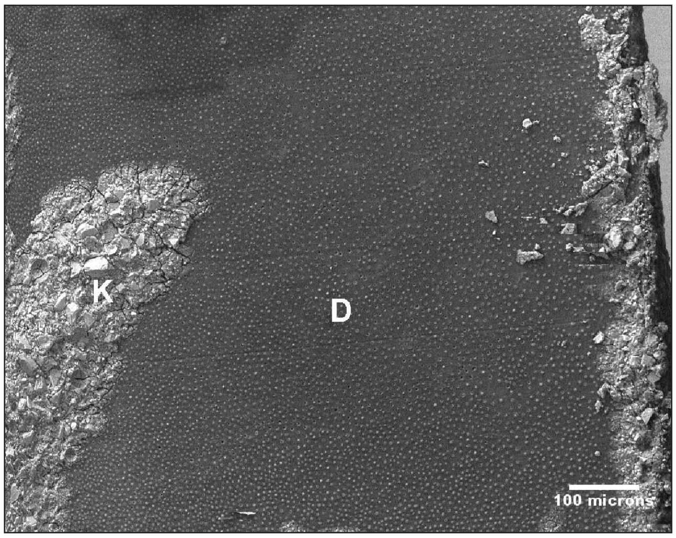

Figure 4. *Mixed failure specimen in the Ketac-Fil/Carious dentin group. K: Cohesively failed region within Ketac-Fil. D: Adhesively failed region between Ketac-Fil and dentin. Dentin surface was exposed.*

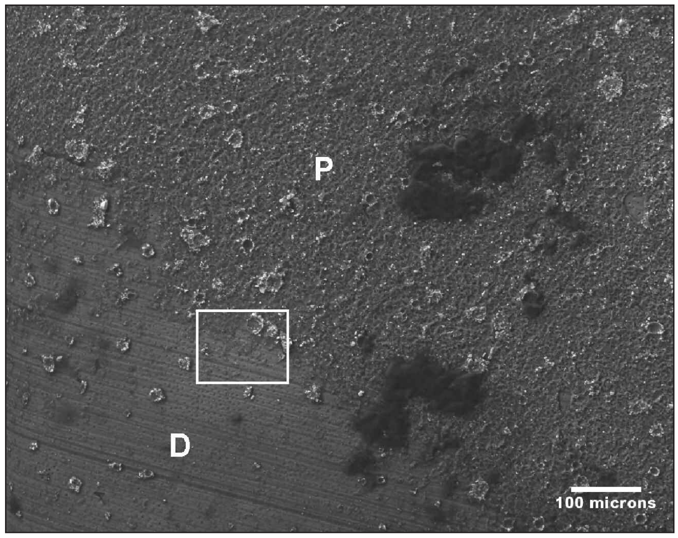

Figure 5. *Mixed failure specimen in the Photac-Fil/Sound dentin group. P: Cohesively failed region within Photac-Fil. D: Adhesively failed region between Photac-Fil and dentin.*

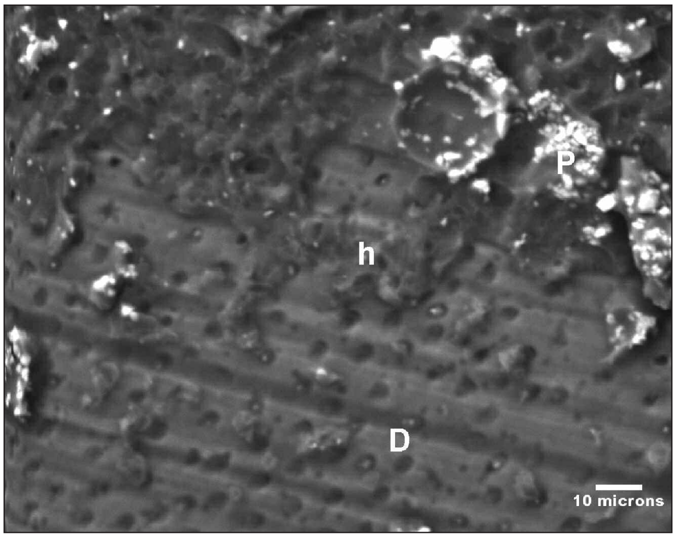

Figure 6. *High magnification image of indicated area in Figure 5. h: A layer is covering the bare dentin. D: The adhesively failed region between Ketac-Fil and dentin.*

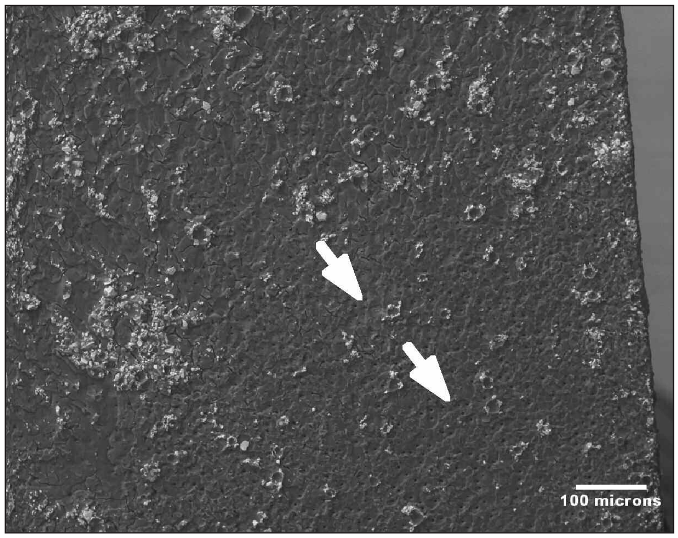

Figure 7. *SEM image of a specimen that was determined a cohesive failure under light microscopy in the Photac-Fil/Sound dentin group. Exposed dentinal tubules (white arrows) in many small areas are seen under SEM.*

Figure 4 shows the features of the mixed failure mode in the Ketac-Fil/Carious Dentin group. Many occluded and opened dentinal tubules with exposed dentin surface are seen in this image.

Figure 5 shows a specimen with mixed failure from the Photac-Fil/Sound Dentin group; Figure 6 is a higher magnification of the area indicated in Figure 5. Figure 7 shows a typical cohesive failure from the same group. This specimen was determined to be a cohesive failure under light microscopy (LM). However, exposed dentinal tubules are seen in many areas under SEM. Figure 8 is a higher magnification of one of these areas. A layer partially covering the dentin surface is seen between Photac-Fil and dentin.

Figure 9 shows a cohesive failure in the Photac-Fil/Carious Dentin group. Similar to cohesive failure in the Photac-Fil/Sound Dentin group, this specimen was considered to be a cohesive failure under LM. However, SEM showed evidence of dentinal tubules in many small areas. Figure 10 shows a mixed failure in the same group. Fractured Photac-Fil surface, a thin layer located between Photac-Fil and dentin, and bare dentin are visible in this figure.

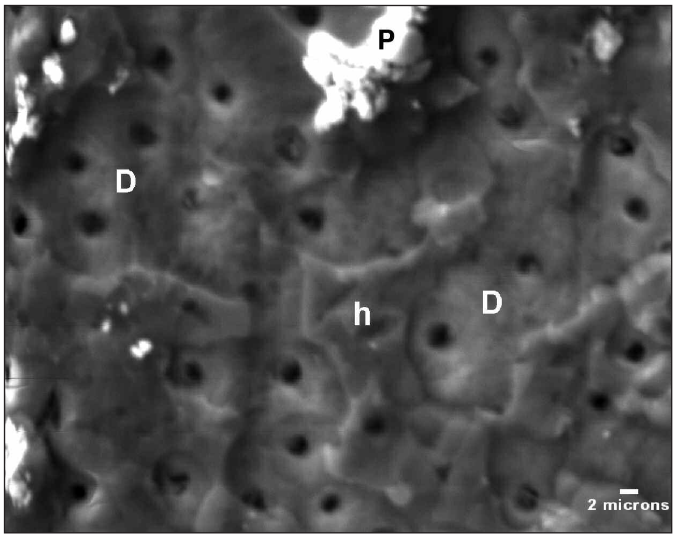

Figure 8. *Higher magnification image of a specimen in the same group as Figure 7. P: Cohesively failed region within Photac-Fil. D: The adhesively failed region between Photac-Fil and dentin. h: A layer covering dentin under Photac-Fil.*

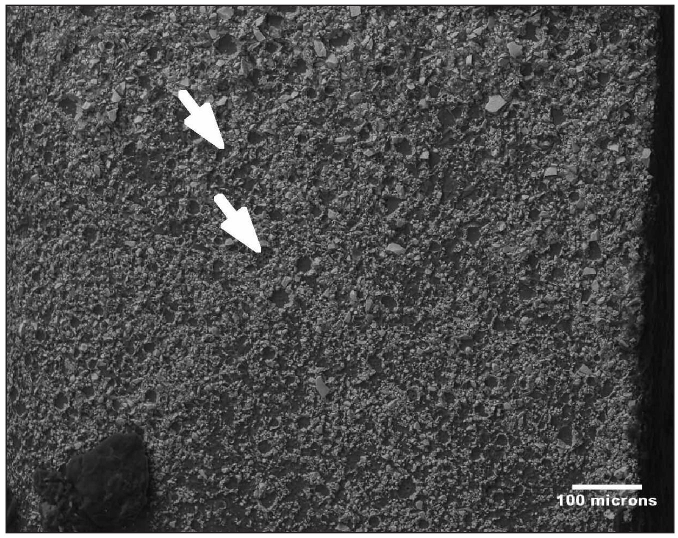

Figure 9. *SEM image of a specimen determined a cohesive failure under light microscopy in the Photac-Fil/Carious dentin group. Exposed dentinal tubules (white arrows) in many small areas are seen under SEM.*

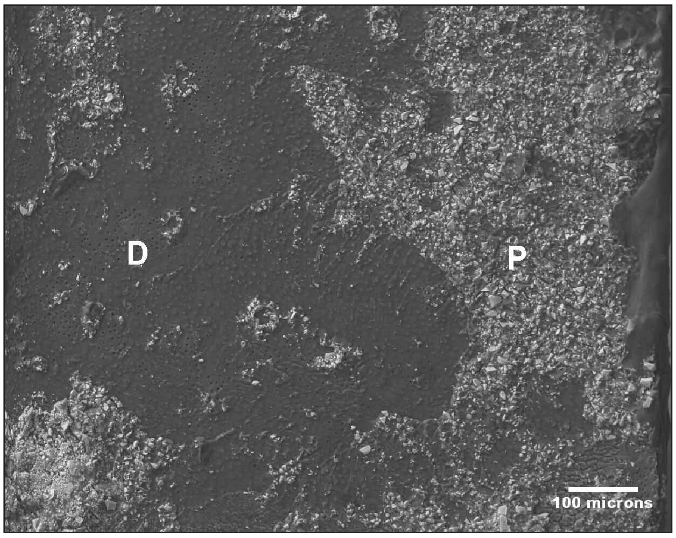

Figure 10. *Mixed failure specimen in the Photac-Fil/Carious dentin group. P: Cohesively failed region within Photac-Fil. D: Adhesively failed region between Photac-Fil and dentin.*

Failure Mode Analysis

Failure mode classification and fractographic analysis of the debonded specimens have been important parts of bond strength testing. This process includes the analysis of the correlation between bond strength and failure mode. In this study, the most prominent failure mode of both GICs to sound dentin was cohesive failure within the glass ionomer material. This observation was consistent with many previous studies.[18-21] However, it is different from studies using the same methodology.[22-23]

The predominance of a cohesive failure mode of glass ionomer materials to sound dentin has often been interpreted as showing that the bond strength values represent a lower tensile strength of the tested GIC material rather than its true adhesive bond strength to dentin. To examine this hypothesis, the microtensile strengths of Ketac-Fil and Photac-Fil were measured using the same test method as the bond strength test except that hour glass-shaped specimens were prepared instead of beam-shaped specimens. In this measurement, the mean microtensile strength of Ketac-Fil was 16.9 ± 2.9 MPa (n=12), and the mean microtensile strength of Photac-Fil was 39.3 ± 11.8 MPa (n=12). The mean cross-sectional area of the specimens was 0.50 mm^2 for Ketac-Fil and 0.48 mm^2 for Photac-Fil. Because the mean microtensile bond strength value of the cohesively failed Ketac-Fil specimens was 14.04 ± 2.48 MPa, the difference between the microtensile bond strength and the microtensile strength of the material was minute in Ketac-Fil. However, the difference between the microtensile strength and the microtensile bond strength was significant in Photac-Fil (39.3 MPa versus 22.5 MPa). These results indicate that the cohesive failure mode may represent tensile failure of the material itself for Ketac-Fil; but this is not true in Photac-Fil, because of the considerable difference between the microtensile bond strength and the microtensile strength of Photac-Fil.

Another issue is whether the cohesive failure mode means that the true adhesive bond strengths (forces) of either material is stronger than the cohesive strength of the material itself. To investigate this question, the theory of cohesive versus adhesive separation in an adhesive system by RJ Good was adopted to this study.[24]

Employing the Griffith-Irwin crack theory, Good proposed the important parameters in separation of a 2-phase adhering system. The important parameters are the difference in elastic moduli (ΔE), the difference in energy dissipation per unit crack extension (ΔG), the thickness of the region where dissipation occurs (δ_1 or δ_2) and the presence or absence of strong interfacial bonds. Analyzing these parameters in the formulation of the Griffith-Irwin crack theory of fracture, the most probable location for failure initiation can be determined as being within one phase or at the interface. In this analysis, if ΔE and ΔG have the same direction, the fracture in a 2-phase adhering system may occur within the phase, which has the lower elastic modulus (E) and lower energy dissipation per unit crack extension (G), even though adhesive forces at the interface are weaker than cohesive forces in the bulk phases. In addition, true interfacial failure can be predicted only if the adhesive forces at the interface are very weak. If ΔE and ΔG have the same direction, and if interfacial forces are strong, then the most probable site for failure initiation is within one phase. These situations can be applied to GIC-dentin bonding systems, because ΔE and ΔG have the same direction in dentin and GIC.[25-26] Therefore, the cohesive failure mode in the GIC-dentin bonding systems does not always mean that the true adhesive bond strengths (forces) of the materials is higher than the cohesive strength of the materials theoretically.

The third issue to consider is the vast difference between microtensile strengths of the Photac-Fil specimens and microtensile bond strengths of cohesively failed specimens in the Photac-Fil/Sound dentin group. This discrepancy may be explained through SEM observation. In the Ketac-Fil group, cohesive failure occurred completely within Ketac-Fil, in most cases, apart from the interface (Figure 2). However, the Photac-Fil group showed a different pattern of failure. Even though the specimens were classified as cohesive failures using light microscopy, high magnification under SEM showed that it was actually a mixed failure mode between the material and dentin (Figures 7 and 8). These different features of the failure pattern in the same cohesive failure mode classification may explain the significant difference in microtensile bond strength and microtensile strength in Photac-Fil.

The predominant failure mode of both Ketac-Fil and Photac-Fil to carious dentin was mixed failure between the GIC and carious dentin. This result appears similar to a study by Palma-Dibb and others.[15] It seems that the difference of this prominent failure mode between the sound dentin and carious dentin groups may be related to the considerably lower bond forces of both materials to carious dentin. In Good's separation theory,[24] if ΔE and ΔG have the same sign, true interfacial failure can be predicted only if the adhesive forces at the interface are very weak.

Artificial Caries Creation

This study used artificially created carious dentin instead of natural carious dentin. Artificial carious lesions have advantages in making flat bonding surfaces and in standardizing the degree of demineralization of the dentin. Several methods have been introduced to produce artificial carious lesion.[27-30] Because not one of these papers provides comprehensive information on the degree of demineralization and depth of lesion in dentin, the method described by Toro-Arrivillaga was adopted for this study. According to the analysis using transverse microradiography by Toro-Arrivillaga, the methodology used in this study has created an average 70.4 ± 16.5 µm depth of carious dentin and 1511.0 ± 326.8 Vol % x µm of mean mineral loss (ΔZ).[11]

In this study, the correlation between the degree of demineralization in dentin and the bond strength of GICs to carious dentin is not clear. Further research is needed on this topic.

Premature Failure of Specimens

A total of 13 specimens were broken during specimen preparation. Six specimens failed during the sawing procedure, and 7 specimens failed during cutting of the dentin base. The number of premature failures was 1 in the Ketac-Fil/Sound dentin group; 7 in the Ketac-Fil/Carious dentin group; 1 in the Photac-Fil/Sound dentin group and 4 in the Photac-Fil/Carious dentin group. The high number of premature failures in the carious groups seems to reflect the weak bond strength of both groups. The bond strength value of all premature failed specimens was considered to be 0. Two specimens, which broke accidentally during handling in the Photac-Fil/Carious dentin group, and one specimen that had Zapit flow on the interface of bonding in the Photac-Fil/Sound dentin, were excluded from all calculations.

CONCLUSIONS

This study led to the following conclusions and observations:

1. The microtensile bond strengths of Ketac-Fil Plus Aplicap and Photac-Fil Aplicap to carious dentin were significantly lower than those to sound dentin.
2. The microtensile bond strength of Photac-Fil Aplicap to both sound and carious dentin was significantly higher than that of Ketac-Fil Plus Aplicap.
3. There was a significant difference in failure mode between the sound dentin and carious dentin groups. In the sound dentin groups, cohesive failure was predominant; whereas,

mixed failure was predominant in the carious dentin groups.

4. In the Photac-Fil/Sound Dentin group, the specimens determined to be cohesive failures under light microscopy showed mixed failure under SEM.

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