Effect of adhesive liner on the shear bond strength of indirect restoration to enamel and dentin

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Purpose: This study investigated whether the bonding of a resin inlay to enamel and dentin could be improved by applying both an adhesive system and a low-viscosity microfilled resin to the underlying tooth substrate.

Materials and Methods: A self-etching primer system (Clearfil SE Bond) and a low-viscosity microfilled resin composite (Protect Liner F) was applied to one half of enamel or dentin slices, with the remaining slices served as non-lined specimens, with only Clearfil SE Bond applied. Composite inlays (Estenia) were fabricated indirectly and cemented with a dual-cured resin cement (Panavia Fluoro Cement). After 24-hour storage in 37˚C water, the bonded inlays were subjected to a micro-shear bond test, whereby a shear force was applied to the inlays. The data were compared with observations of the directly placed controls (Clearfil SE bond and Clearfil AP-X); statistical analysis was carried out using ANOVA and Fisher's PLSD test. Morphological observations using confocal laser scanning microscopy were also performed.

Results: The enamel surfaces did not show any a statistical difference between the shear bond strength values observed in the lined and non-lined groups ($p>0.05$). However, the bonding capacity of Estenia inlay with the liner to dentin was slightly higher than that of the non-lined restoration. The bonding of indirect restorations to enamel was almost the same as that of the directly placed resin composite ($p>0.05$).

Conclusion: The resin liner might improve the bonding of resin cement to dentin, whereas no effect was observed on enamel. The results of the present study suggest that, from the perspective of shear stress, it seems advantageous if an adhesive layer on the cavosurface enamel were thin rather than thick.

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Clinical Significance: Using a low-viscosity resin in indirect restorations showed an improvement in bonding of resin cement to dentin, whereas no significant difference was seen in the case involving of enamel.

Key Words: low-viscosity microfilled resin, micro-shear bond strength, self-etching primer.

Introduction

Indirect adhesive procedures are being used more widely for esthetic restorations. Metal and metal-free inlays, veneers, crowns, resin-bonded fixed prostheses, and even posts are now routinely bonded to tooth structures via the use of resin cement.[1-9] On the other hand, evidence of insufficient marginal sealing of bonded restorations has also been shown in microleakage studies.[9,10] A clinical study using bonded inlays reported the incidence of post-cementation sensitivity as high as 30%.[11] The application of dentin bonding systems to the prepared cavity wall has been advocated to prevent postoperative sensitivity in cases involving indirect restoration of vital teeth.[4]

Recently, application of a dentin bonding agent and low-viscosity microfilled resin on a prepared dentin surface has been advocated for indirect restorations.[12,13] It has been reported that the application of low-viscosity microfilled resin could reduce the influence of polymerization shrinkage of resin composite and resin cements.[14,15] Although many studies have focused on the bonding of resin cement to enamel, dentin, or indirect restorative materials, little is known about the bonding of indirect restorative materials to the tooth structure via the use of resin cement.[1-9] This lack of information is probably due to the difficulties of fabricating samples and designing a simple method of testing two adhesive interfaces (i.e., one interface between the tooth and the resin cement, and the other between the indirect restoration and the resin cement).

Recently, a micro-shear bond test was developed that enabled measurement of bonding of adhesive materials to a variety of tooth substrates; the method allows for ease of sample preparation and gives precise results with relatively small standard deviations.[8,16] The aim of this study was to determine whether the bonding of a resin inlay to enamel or dentin could be improved by applying an adhesive system and low-viscosity microfilled resin. Bond strengths were assessed by means of a micro-shear bond test, and the failure mode was observed using confocal laser scanning microscopy (CLSM). CLSM has been widely used in biology for noninvasive and nondestructive imaging *in vivo* of many organ tissues and has found numerous applications in dental research.[17] Specimens examined by CLSM do not require any special preparation and are not subjected to the distortions caused by dehydration that results from procedures such as scanning electron microscopy.

Materials and Methods

Tooth Preparation

Bonding was performed on buccal surfaces of enamel and dentin surfaces of extracted human molars that were stored at 4°C in saline. Sixty teeth were used in this study. Thirty enamel and dentin slices, each approximately 1.0 mm thick, were cut parallel to the buccal surface from the mid-coronal region using a slowly rotating diamond blade (Struers Minitom, Struers, Copenhagen, Denmark) and water lavage and wet 600-grit SiC paper was used to create flat surfaces with a uniform smear layer thickness. The enamel or dentin surfaces were treated with Clearfil SE Bond Primer (Kuraray Medical, Inc., Tokyo, Japan, Lot # 00295A) for 20 s, dried, Clearfil SE Bond (Kuraray Medical, Inc., Lot #00364A) applied, air-thinned, and light-cured for 10 s. Prior to irradiation of the bonding resin, an iris that was cut from micro bore tygon tubing (R-3603, Norton Performance Plastic Co., Cleveland, OH, USA) with an internal diameter and a height of approximately 0.8 mm and 0.5 mm respectively (Fig. 1), and mounted on the enamel or dentin slice to delineate the bonding area.

Preparation of Composite Resin Inlay

A cylinder from the micro bore tygon tubing (R-3603) with an internal diameter of 0.8 mm and a height of 0.5 mm was cut and used as a mold for the composite resin inlay (Fig. 1). A resin composite for indirect restorations (Estenia, Shade DA2, Kuraray Medical Inc., Lot #00209B) was placed into the tubing on a flat surface covered with a paper mixing pad, a clear plastic matrix strip was placed over the resin, gently pressed flat and irradiated for 60 s using a visible light curing unit for laboratory (α-Light II, J. Morita Co., Tokyo, Japan), prior to heat curing at 110˚C for 15 minutes in air (KL 100, Kuraray Medical, Inc.). Very small cylinders of resin inlay, approximately 0.8 mm in diameter and 0.5 mm high, were removed from the tygon tubing and silanized with the application of a mixture of acidic primer (Clearfil SE Bond Primer) and a silane agent (Clearfil porcelain bond activator, Kuraray Medical, Inc., Lot #00128A) for 20 s.

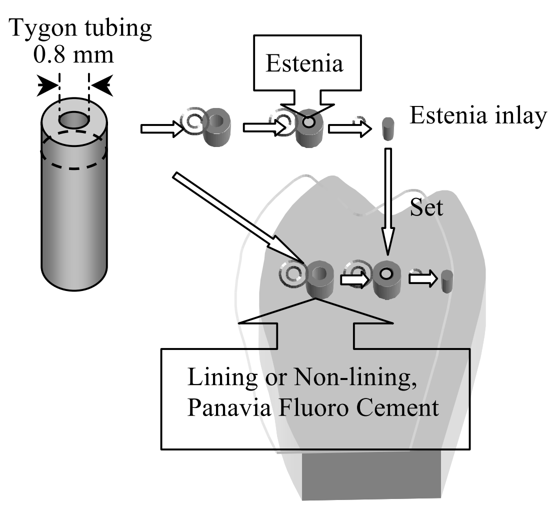


Fig. 1. Bonding procedure for Estenia inlay.

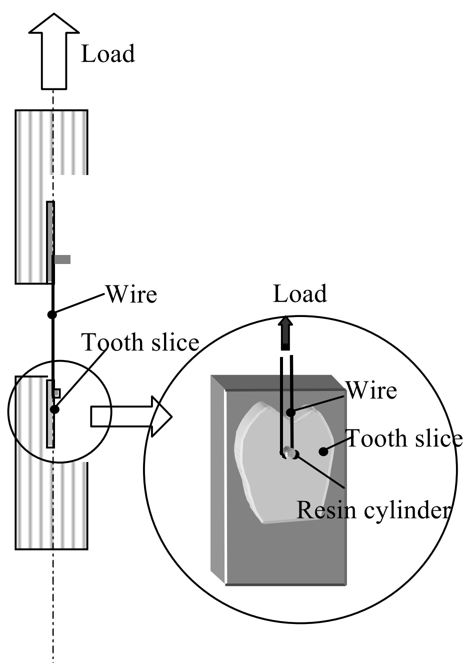


Fig. 2. Schematic diagram of the micro-shear bond test apparatus.

Bonding of Composite Resin Inlay

Thirty enamel slices and thirty dentin slices with tygon tubing were divided and assigned to three treatment sequences (10 enamel or dentin specimens each), and then each specimen received one of the following treatments:

Group 1 (Estenia, non-lining): Dual-cured resin cement (Panavia Fluoro Cement, Kuraray Medical, Inc., Lot A paste: #00158A, B paste: #00090A) was mixed and injected into the tubing on the tooth surface. The small cylinder of resin inlay was then inserted into the tubing, bonded and light cured for 30 s.

Group 2 (Estenia, lining): A low-viscosity microfilled resin (Protect liner F, Kuraray Medical, Inc., Lot #0052) was applied to the tooth surface inside the iris of the tubing and was light cured for 20 s, prior to the injection of mixed Panavia Fuoro Cement into the tubing. The small cylinder of resin inlay was then inserted into the tubing, bonded and light cured for 30 s.

Group 3 (resin composite): A hybrid restorative resin composite, shade A3 (Clearfil AP-X, Shade A3, Kuraray Medical, Inc., Lot #00765A) was placed into the cylinder, and a clear plastic matrix strip was placed over the resin; the specimen was then gently pressed flat and irradiated for 30 s.

In this manner, very small cylinders of resin inlay or resin composite were bonded to the surface. The specimens were stored at room temperature (23˚C) for 1 hour prior to removal of the tygon tubing. All specimens were placed in 37˚C water for 24 hours.

Micro-Shear Bond Test

The micro-shear bond test apparatus is shown in Fig. 2. The tooth slice, with the resin cylinder, was attached to the testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA, USA), which in turn was placed in a Universal testing machine (EZ-test-500N, Shimadzu, Kyoto, Japan) for shear bond testing. A thin wire (diameter: 0.20 mm) was looped around the resin cylinder making contact with half of the cylinder base, and the wire was held flush against the resin/tooth interface. A shear force was applied to each specimen at a crosshead speed of 1.0 mm/minute until failure occurred.

Ten specimens from each group were tested. The data were statistically analyzed using two-way analysis of variance (ANOVA). After two-way ANOVA, one-way ANOVA and multiple comparisons were carried out using Fisher's PLSD test. The bonding methods (i.e., Estenia with no lining, Estenia with lining, or resin composite) and tooth substrates (enamel or dentin) served as the two factors. Statistical significance was defined as $p<0.05$.

All of the debonded tooth surfaces were examined with an optical microscope at 30x magnification and CLSM (1LM21H/W, Lasertec Co., Yokohama, Japan) to determine the mode of failure. Failure modes were categorized into one of six types: A: 100% cohesive failure of the adherend materials, resin cement, resin inlay, resin liner, or resin composite; B: 100% adhesive failure between tooth substrate or hybrid-like layer and adhesive resin; C: 100% cohesive failure in the tooth substrate; AB: Mixed failure with cohesive failure of the adherend materials (A) and adhesive failure (B); BC: Mixed failure with adhesive failure (B) and cohesive failure in the tooth substrate (C); ABC: Mixed failure with cohesive failure of the adherend materials (A), adhesive failure (B), and cohesive failure in the tooth substrate (C). The modes of failure were analyzed using the Kruskal-Wallis test, with significance defined as $p<0.05$.

Results

The mean shear bond strength values (MPa), standard deviations, and the modes of failure are shown in

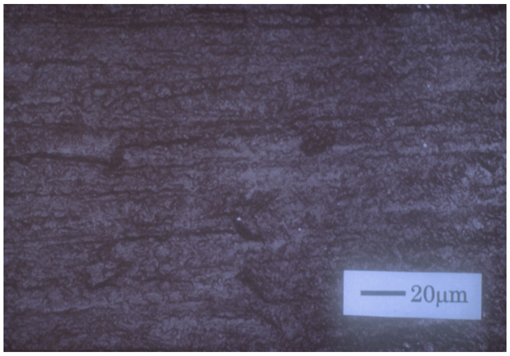
Table 1. The ANOVA indicated that there was a statistically significant variance among the mean bond strength values (two-way ANOVA, F=5.007, p=0.0101). Statistical significance was observed among the shear bond strengths of the indirect restorative materials that were bonded to dentin (one-way ANOVA, F=12.636, p=0.0001), whereas no significance was found among the specimens bonded to enamel (one-way ANOVA, F=1.701, p=0.2006). When the indirect and direct restorations were compared, the bonding of the indirect restorative materials to dentin was found to be significantly lower than that of the direct restorations (Fisher's PLSD test, $p<0.05$). The bonding of Estenia with lining to dentin gave an intermediate bond strength that was also significantly different from that of the direct-restoration specimens (Fisher's PLSD test, $p<0.05$).

Table 1. Results of the micro-shear bond test.

Group	Shear bond strength (MPa)		Failure mode					
	Mean	SD	A	B	C	AB	BC	ABC
Enamel								
Estenia, non-lining	39.6	7.79	0	8	0	0	1	1
Estenia, lining	36.7	5.12	1	6	0	1	2	0
AP-X	41.9	5.39	0	10	0	0	0	0
Dentin								
Estenia, non-lining	29.1	8.26	0	6	1	2	1	0
Estenia, lining	33.7	5.26	1	1	0	7	1	0
AP-X	43.0	6.71	4	2	0	4	0	0

Sample size=10 for each group. SD: Standard deviation.

CLSM observation also revealed that the mode of failure after the shear test was significantly different among the groups (Kruskall-Wallis, p=0.0013). For the three groups on enamel surfaces, a substantial percentage of the failure modes was adhesive failure along the enamel/resin cement or adhesive liner interface (Type B failure, Fig. 3); no significant difference was observed among the enamel surfaces (Kruskall-Wallis, p=0.7049). However, in the case of dentin, various failure patterns were observed (Kruskal-Wallis, p=0.0063). When the dentin surface was coated with low-viscosity resin prior to the bonding of the Estenia inlay, failures were predominantly mixed (i.e., cohesive within dentin and adhesive along the dentin/resin cement or adhesive liner interface; Type AB failure, Fig. 4). Without the liner in the Estenia inlay, adhesive failure (Type B failure) was seen in most cases. Direct composite resin underwent Type B or Type AB failure.



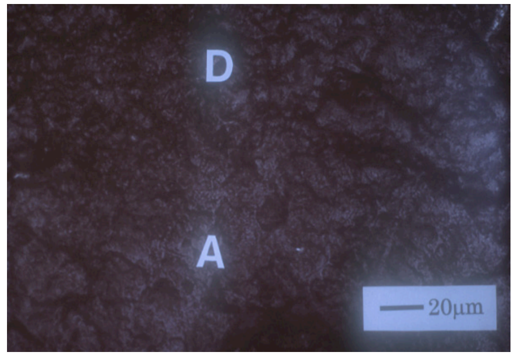


Fig. 3. CLSM image of a debonded enamel surface after the shear bond test. Estenia inlay with no-lining. Adhesive failure below the bonding resin and enamel can be seen (left).

Fig. 4. CLSM image of a debonded dentin surface after the shear bond test. Estenia inlay with resin lining. Mixed failure with adhesive failure (A) and dentin failure (D) can be seen.

Discussion

The hardness and bonding of dual-cured resin cement have both been reported to be low under conditions of self-cure alone without light irradiation.[2,6,8] Moreover, the intensity of the light penetrating through the indirect restorative materials has been shown to be reduced by thickness and shade.[18,19] Considering these factors, it becomes clear that a curing unit must be able to effectively transmit light through an indirect restoration in order to obtain sufficient hardness and bonding of the resin cement immediately after cementation.[5,19] In this study, the light from the curing unit was able to transmit through the Estenia inlay, with a thickness of 0.5 mm, to the resin cement. Tashiro et al.[19] reported that a 1-mm thick Estenia inlay did not adversely affect the bonding of a photo-irradiated dual-cured resin cement. Since the bond strength of resin cement through the Estenia inlay obtained for the enamel was similar to that of the direct resin composite filling, this finding appears to be correct.

Composite restorative materials undergo significant volumetric shrinkage when polymerized.[20] Although the use of indirect composite inlays significantly reduces polymerization shrinkage during placement, contraction of the luting resin composite may also remain problematic.[9,10,21] The usage of elastic materials as a liner during the early stage of setting has been reported to relieve this contraction stress.[22] Jayasooriya et al.[9] observed gap formation under an MOD inlay, and reported that the percentage of the length of the gap formation with the resin liner was significantly lower at the internal dentin-restoration interface compared with that of non-lined specimens. It is likely that the resin liner decreases the microleakege of resin cement bonded to the dentin surface.[9,15,19] In the present study, when Protect liner F was applied to the dentin surface, the bonding of the Estenia inlay was significantly improved, i.e., 33.7 MPa. This finding was in accordance with previous studies.[9,15,19] An explanation for these findings remains to be determined, but it is possible that the additional application of a low-viscosity resin composite might have promoted the polymerization of the adhesive, due to the double-curing of the adhesive and air inhibition at the adhesive surface.[3]

Several clinical and laboratory investigations have revealed that the luting space is the weakest part of indirect restorations.[11,21] Secondary caries following occlusal wear and marginal leakage has been reported, which always occurs at this location.[23] Consequently, the film thickness of the resin cement at the cavosurface enamel margin needs to be thin in order to prevent secondary caries.[9] In this study, the enamel bonding of the Estenia inlay did not improve with the application of Protect Liner F ($p>0.05$). Although no statistical significance was noted between the bond strength values obtained from enamel surfaces, the application of micro-filled resin slightly decreased the bonding of indirect restorative materials (non-lined, 39.6 MPa; lined, 36.7 MPa). In addition, the mode of failure was also similar, and no statistical significance was seen among the groups ($p>0.05$). Previous studies have shown that the film thickness of low-viscosity resin composite and resin cement was over 200 µm, which was obviously thicker than that obtained with the application of bonding resin alone.[9] In this study, the film thickness of low-viscosity resin composite or resin cement might also be thick because of the smallness of specimen dimension. It would therefore be reasonable to speculate that lining with the micro-filled resin composite is not necessary in the case of enamel surfaces; the application of the adhesive only might be more beneficial. However, the usage of a thinner liner might also be an alternative in cases involving enamel margins.

Comparing the bonding of indirect composite restorative materials with that of direct resin composite materials, no significant difference was seen in the specimens involving enamel bonding ($p>0.05$), whereas the bond was significantly reduced when the resin inlay was bonded directly to the dentin surfaces ($p<0.05$). Consequently, the findings indicated that in terms of bonding, direct composite restorations would be the preferred treatment over indirect composite restorations. Furthermore, direct composite restorations require minimal intervention and also minimal cavity preparation.[24] However, a major limitation of direct composites should be noted in this context, namely, they are associated with the inability to control both polymerization shrinkage and the depth of cure.[25] Therefore, for larger restorations, indirect methods are superior alternatives to direct resin composite fillings. With regard to tooth-colored inlays and onlays, factors affecting overall restoration longevity may be related to luting and finishing procedures, and may be dependent on the width and performance of the resin composite lute.[4,11,21] Furukawa et al.[5] reported that reinforcement by the unification of the tooth substrate and the indirect restoration was achieved if the bonding of resin cement to the tooth substrate was adequate. Even though the application of the low-viscosity resin improved the bonding of the resin cement to the dentin surface, the capacity for bonding was still lower than that of the direct composite ($p<0.05$; Estenia, resin lining, 33.7 MPa; AP-X, 43.0 MPa). Development of new luting materials or methods that can effectively bond both to tooth and to the indirect restorative materials will most likely be necessary in the future.

The cervical enamel/composite interface and the dentin/composite interface have repeatedly been reported to be more vulnerable to microleakage than the incisal enamel/composite interface *in vitro*.[10] Even at the enamel margin, the enamel structure of the cervical border frequently exhibits a disturbed prism arrangement lacking a normal keyhole appearance; the bonding capacity of this margin has been reported to be low.[16] Again, further investigation will be necessary to develop bonding resins and cements that produce high, uniform bond strength and/or gap-free margins in cases involving indirect restorations.

Conclusion

In spite of the limitations of this laboratory study, it was concluded that resin lining with a low-viscosity composite was necessary when indirect resin composite restorative materials were bonded to a dentinal surface. In cases involving an enamel surface, the influence of the low-viscosity composite on the bonding of indirect restorative materials was unclear. As the performance of the materials was not considered in a clinical setting, further investigation using clinical trials are necessary.

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