Original Article

Microtensile Bond Strength of Indirect Resin Composite to Resin-coated Dentin: Interaction between Diamond Bur Roughness and Coating Material

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Abstract

This aim of this study was to determine the effect of type of bur and resin-coating material on microtensile bond strength (μ TBS) of indirect composite to dentin. Dentin surfaces were first ground with two types of diamond bur and resin-coated using UniFil Bond (UB) or Adper Single Bond (SB), and then bonded to a resin composite disc for indirect restoration with adhesive resin cement. After storage for 24 hr in distilled water at 37°C, μ TBS was measured (crosshead speed 1 mm/min). When UB was applied to dentin prepared using the regular-grit diamond bur, μ TBS was significantly lower than that in dentin prepared using the superfine-grit bur. In contrast, no significant difference was found between regular-grit and superfine-grit bur with SB. However, more than half of the superfine-grit specimens failed before μ TBS testing. These results indicate that selection of bur type is important in improving the bond strength of adhesive resin cement between indirect resin composite and resin-coated dentin.

Key words: Diamond bur roughness—Resin coating—Microtensile bond strength—Indirect composite restoration

Introduction

To achieve a prepared cavity surface with a hybrid layer and tight sealing film, a resin-coating technique was proposed for indirect restorations in the early 1990's which employed

an adhesive system and low-viscosity resin composite^{18,24}. This technique has recently been shown to be capable of not only increasing the bond strength of resin cement and producing good interfacial adaptation^{7,8,10,17}, but also protecting the prepared dentin

and underlying vital pulp tissue^{16,27}, thereby allowing minimally invasive restoration in its clinical application¹⁹.

The success of any approach to direct or indirect restoration tends to depend on adhesive performance. However, several other factors are also considered clinically important, including regional differences in dentin (dentin depth and/or presence of dentin tubules)^{1,25,29}, cavity configuration³, temperature and relative humidity^{2,15}, dentin wettability⁴, saliva/blood contamination^{5,36}, storage condition of adhesive system^{6,20}, and degree of conversion and strength of the adhesive material itself^{8,31}. Type of bur has also been reported to affect bond strength^{12,21,22,28}. Differences in bond strength due to type of bur arise from the thickness of the smear layer rather than the roughness of the dentin itself^{13,32-34}. These differences have been reported in both direct and indirect bonding¹¹.

With indirect restoration, further factors may affect bond strength due to the greater number of steps involved. These include differences in adhesive system, application of low-viscosity resin composite immediately after light curing of adhesive resin, influence of impression and temporary filling material, and resin-based luting cement. Differences in type of bur may also affect bond strength in both direct and indirect dentin bonding.

The purpose of this study was to determine the effect of type of bur on the microtensile bond strength (μ TBS) of a 2-step etch & rinse adhesive and a 2-step self-etch adhesive to resin-coated dentin. The null hypothesis tested in this study was that bur selection would yield no difference in μ TBS between resin-coated dentin and indirect resin composite bonded with adhesive resin cement.

Materials and Methods

1. Test teeth

Twenty-six caries-free human molars collected with informed consent under a protocol reviewed and approved by the Commission for Medical Ethics of Tokyo Dental College

Chiba Hospital (No.153) were stored in an aqueous solution of 0.5% Chloramine-T at 4°C and used within 3 months of extraction. The coronal surfaces of the teeth were trimmed using a model trimmer (MT-7, J. Morita Tokyo Mfg. Co., Tokyo, Japan) to form a long, flat dentin surface at the mid-coronal portion.

Each surface was flattened by hand with 600-grit silicon carbide paper under running water, and then ground with a regular-grit diamond bur (Diamond Point FG, #114, Shofu, Kyoto, Japan; mean diameter of diamond particle = 100 μ m) connected to a 1:5 high-speed motor handpiece (INTRAmatic LUX-3, KaVo, Biberach, Germany). Fourteen dentin surfaces were further prepared with a superfine-grit diamond bur (Diamond Point FG, #SF 114, Shofu; mean diameter of diamond particle = 25 μ m). The two types of dentin substrate were randomly assigned to one of three groups: two coated adhesive groups and a non-coated control group. Four teeth were assigned to all groups, except the Adper Single Bond/Superfine-grit group, to which a further 2 teeth were added (total of 6 teeth) as the number of pre-tested failures was larger than in the other subgroups (subscribed below).

2. Resin materials and bonding procedures

Materials used in this study are listed in Table 1. For indirect restoration resin coating, a 2-step self-etch adhesive (UniFil® Bond, GC, Tokyo, Japan) and a 2-step etch & rinse adhesive (Adper™ Single Bond, 3M ESPE, St. Paul, MN, USA) were used with a flowable resin composite, UniFil Flow (A3 Shade, GC). Linkmax™ was used as the resin cement (Clear shade, GC).

To prepare indirect composite discs, Gradia® (Incisal shade, GC) was poured into an acrylic ring 9 mm in diameter and 5 mm in height, pressed with two slide glasses, pre-cured with New Light VL-II (GC) for 20 sec each side, and then cured in Triad® II (Dentsply International, York, PA, USA) for 10 min (5 min each side). After polymerization, the Gradia slabs were sand-blasted with 50- μ m alumina and

Table 1 Materials used in this study

Material	Manufacturer	Components/Shade (Lot No.)	Principal ingredients	Procedure	pH
[Adhesive] UniFil Bond	GC, Tokyo, Japan	Primer (0507201)	HEMA, Ethanol, 4-MET	Apply primer (20sec.), air dry, apply bonding agent, light cure (10sec.)	2.0
		Bonding agent	UDMA, HEMA, CQ		
Adper Single Bond	3M ESPE, St Paul, MN, USA	Etchant (6KT)	Phosphoric acid	Apply etchant (15sec.), rinse with water spray (10sec.), gentle air dry (1sec.), apply two consecutive coats of adhesive with fully saturated brush tip, gentle air dry (5sec.), light cure (10sec.)	>1 4.3
		Adhesive (6KP)	HEMA, water, ethanol, amines, bis-GMA, DMA, copolymer of polyacrylic and polyitaconic acids, CQ		
[Flowable composite] UniFil Flow	GC, Tokyo, Japan	A3 shade (0610171)	Fluoro-Alumino Silicate Glass, Di-2-Methacryloyloxyethyl 2,2,4-trimethylhexamethylene dicarbamate, Silica, TEGDMA	Apply with brush-tip, light cure (20sec.)	
[Resin adhesive luting cement] Linkmax	GC, Tokyo, Japan	Self-etching primer A (0610071)	water, 4-MET, ethanol, methacryloyloxyester		1.5 2.0 (A+B)
		Self-etching primer B (0610071)	ethanol, catalyst		6.0
		Paste A/B (clear shade) (0610133)	fluoro-alumino silicate glass, HEMA, UDMA, methacryloyloxyester, silica,		
		Composite primer	HEMA, Di-2-Methacryloyloxyethyl, 2,2,4-trimethylhexamethylene	Coat composite primer to bonded Gradia surface with brush-tip, light cure (20sec.)	
[Indirect composite] Gradia	GC, Tokyo, Japan	Translucent shade	Alumino-silicate glass, Amorphous Precipitated Silica, Di-2-Methacryloyloxyethyl 2,2,4-trimethylhexamethylene dicarbamate, Neopentylglycol dimethacrylate	Pre-cure (20sec. for each side) with New Light VL-II, final cure (Triad II, Dentsply International; 5min. each side), sandblast bonded surface with 50 μ m alumina.	

HEMA = 2-hydroxyethyl methacrylate; 4-MET = 4-methacryloyloxyethyl trimellitate; UDMA = urethane dimethacrylate; CQ = champhorquinone; bis-GMA = bisphenol A-glycidyl dimethacrylate; DMA = dimethacrylate; TEGDMA = triethylene glycol dimethacrylate

rinsed with tap water. The sand-blasted surfaces were surface-treated with Composite Primer (GC) and visible light-irradiated with New Light VL-II for 20sec immediately prior to bonding.

A schematic of specimen preparation and μ TBS testing is given in Fig. 1. Each adhesive system was applied to the ground dentin surfaces according to the manufacturer's instructions (see Table 1), followed by immediate application of UniFil Flow with a disposable micro-brush tip and light-curing (New Light VL-II) for 20sec. After wiping off the uncured layer with an alcohol-soaked cotton pellet, the dentin surface was conditioned with a mixture of Linkmax self-etching primer A

and B for 30 sec, and gently air-dried. Immediately after, the Gradia slab was bonded to the dentin surface with Linkmax. Ninety seconds later, the bonded interface was light-cured with New Light VL-II for 20sec, and further built up with a PMMA rod (8 $\times$ 8 $\times$ 3 mm) using 4-META/MMA-TBB resin (Superbond[®] C&B, San Medical, Moriyama, Japan). Each light-curing with New Light VL-II was controlled to approximately 700 mW/cm².

Thirty minutes after cementing, all specimens were stored in water at 37°C for 24hr. Thereafter, each bonded specimen was sectioned perpendicular to the bonded interface to obtain 4 or 5 slabs of 0.7 mm in thickness. Each slab was then trimmed with a superfine

Fig. 1 Schematic of specimen preparation for microtensile bond strength testing

a) sectioning perpendicular to occlusal surface to obtain flat dentin surface by silicone-carbide paper, b) grinding of dentin surface by diamond bur, c) resin-coating to ground dentin surface, d) bonding of indirect resin composite with adhesive resin cement, e) bonding of PMMA rod to indirect composite, f) sectioning of bonded specimen to obtain bonded dentin slabs and trimming of slabs into hourglass shape, and g) microtensile bond testing.

diamond bur (#SF 114, Shofu) to obtain an hourglass shape, so that the narrowest portion at the interface was approximately 1.4 mm. The thickness of the bonded area of each specimen was then verified with a digital micrometer (Mitutoyo, Tokyo, Japan).

Each specimen was attached to a Bencor Multi-T testing apparatus (Danville Engineering Co., San Ramon, CA, USA) with cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Ohtawara, Japan) and placed in a universal testing machine (Tensilon RTC-1150-TSD, Orientec, Tokyo, Japan) for microtensile bond strength (μ TBS) testing at a crosshead speed of 1 mm/min. μ TBS was derived by dividing imposed force (N) at time of fracture by bond area (mm^2). When a specimen failed during processing, μ TBS was set at 0 MPa. Data were analyzed by a one-way and two-way ANOVA at a 5% level of significance.

3. Failure mode analysis

To determine mode of failure, both the dentin and composite halves of the fractured

specimens were observed under a stereomicroscope (MS-803, Moritex, Tokyo, Japan) at $210\times$ magnification. Mode of failure was classified into 5 categories: adhesive interfacial (fracture between dentin or hybrid layer and overlying adhesive); mixed R/AI (mainly cohesive failure in dentin or resin material and partial adhesive interfacial failure in same sample); mixed AI/R (mainly interfacial and partial cohesive failure in dentin or resin material in same sample); cohesive in resin (failure within adhesive, resin cement and/or Gradia); or cohesive in dentin. Most frequent failure mode and μ TBS closest to the mean were determined by field-emission scanning electron microscopy (FE-SEM; JSM-6340F, JEOL, Tokyo, Japan).

Results

The mean (SD) μ TBS, number of specimens (n), and number of pre-testing failures (PTF) in all groups are summarized in Table 2.

Table 2 Mean (SD) μ TBS (MPa), number of specimens (n), and number of pre-testing failures (PTF) for all tested groups

		Mean (SD)	PTF/ n
UniFil Bond	Regular	25.6 (10.8) ^b	1/19
	Superfine	38.1 (18.5) ^a	0/19
Adper Single Bond	Regular	20.1 (13.6) ^{bc}	4/17
	Superfine	10.7 (13.4) ^{cd}	16/28
Non-coated (Control)	Regular	16.4 (8.9) ^{cd}	1/19
	Superfine	19.9 (11.4) ^c	2/20

Same letters represent no statistically significant difference (Tukey-Kramer test; $p > 0.05$).

Fig. 2 Distribution (as percentage) of failure mode according to stereomicroscopic observation R: 100% cohesive in resin; R/AI: mixed, mainly cohesive in resin, and partially at adhesive interface; AI/R: mixed, mainly at adhesive interface, and partially cohesive in resin; AI: 100% at adhesive interface; D: 100% cohesive in dentin.

While the two-way ANOVA revealed that 'adhesive system' had a significant effect on μ TBS ($p < 0.0001$), no significant difference was found between 'type of bur' ($p = 0.5442$). However, both factors revealed a significant interaction in μ TBS ($p = 0.0011$). Therefore, further analysis to determine significant differences between the groups was carried out using the *post hoc* Tukey-Kramer test. Comparison of bur type with UniFil Bond revealed that μ TBS with the superfine-grit bur was significantly higher than that with the regular-grit bur ($p < 0.05$). On the other hand, no significant difference was found between the two bur types with Adper Single Bond

($p > 0.05$). However, more than half of the superfine-grit specimens (16/28) failed during processing. Furthermore, no significant difference was found between regular-grit and superfine-grit in the non-coated controls ($p > 0.05$).

Distribution of failure mode is summarized in Fig. 2, and representative SEM photographs of the fractured surfaces after μ TBS testing in each group are shown in Figs. 3, 4, and 5. With UniFil Bond, the percentage of failure at the coated resin with superfine-grit was higher than that with regular-grit (Fig. 2). Almost all dentinal tubules found at the failed surface were plugged with coated adhesive

Fig. 3 Representative SEM images of fractured surfaces on dentin side after microtensile bond strength test (a) UniFil Bond/regular, 21.8 MPa; (b) UniFil Bond/superfine, 43.2 MPa. Mixed adhesive interfacial area failure and cohesive failure in coated resin are apparent in each diamond-grit. Almost all dentinal tubules were plugged with coated material (white arrows). RC: resin cement; CM: coated material; HL: hybrid layer.

Fig. 4 Representative SEM images of fractured surfaces on dentin side after microtensile bond strength test (a) Adper Single Bond/regular, 23.3 MPa; (b) Adper Single Bond/superfine, 11.2 MPa. In each diamond-grit, failure was observed throughout adhesive interface. Open dentinal tubules are clearly visible (white arrows). Black arrows: scratches caused by preparation with diamond bur.

(Fig. 3). With Adper Single Bond, almost all specimens failed at the adhesive interface or showed mixed failure, mainly due to interfacial failure (Fig. 4). The fractured surface at the interfacial site with Adper Single Bond showed open dentinal tubules, unlike with UniFil Bond. With non-coated control, more than half of the specimens showed interfacial failure with both regular-grit and superfine-grit bur preparation, but almost all dentinal tubules were plugged with resin cement (Fig. 5).

Discussion

Several factors have been reported to affect bond strength to resin-coated dentin, namely, the adhesive system itself, combined application of a low-viscosity flowable composite and adhesive system, resin cement, and tooth substrate. In this study, the influence of two factors, type of bur and adhesive system, on μ TBS between dentin and indirect resin composite was determined. In non-coated dentin, no significant difference was found between

Fig. 5 Representative SEM images of fractured surfaces on dentin side after microtensile bond strength test (a) Non-coated/regular, 15.7 MPa; (b) Non-coated/superfine, 20.5 MPa. Mixed adhesive interfacial area failure and cohesive failure in resin cement are apparent in each diamond-grit. Almost all dentinal tubules were plugged with resin cement (white arrows). Black arrows: scratches caused by bur-grinding.

regular-grit prepared dentin and superfine-grit prepared dentin ($p > 0.05$). Linkmax is categorized as a self-etch resin cement, and the acidity of the self-etching primer was mild ($\text{pH} = 2.0$). Koase *et al.*¹¹⁾ also investigated the μTBS of Linkmax to both regular-grit prepared and superfine-grit prepared dentin, and, contrary to the present results, found that superfine-grit produced a significantly higher μTBS than regular-grit. In their study, however, 6 of the 16 superfine-grit prepared dentin specimens failed before μTBS testing, although no regular-grit prepared specimens failed. Since they excluded any data pertaining to pre-test failure when calculating mean μTBS , the present results should not be interpreted as contradicting theirs.

In this study, superfine-grit preparation produced a significantly higher μTBS than regular-grit in UniFil Bond-coated dentin. Dentin abrasive roughness has been reported to influence μTBS in direct bonding of resin composite^{21,22,28)}. The μTBS of regular-grit diamond bur-cut dentin to direct bonded resin composite has been reported to be lower significantly than that of #600 silicon-carbide paper-ground dentin for each self-etch adhesive tested²²⁾. Tay *et al.*³³⁾ reported that the μTBS of direct bonded resin composite with Clearfil® SE Bond (Kuraray Medical, Kurashiki,

Japan) 2-step self-etch adhesive to #60-grit silicon-carbide paper-ground dentin was significantly lower than that to #180- or #600-grit ground dentin. They also demonstrated the presence of a hybridized smear layer immediately above the hybrid layer due to penetration of adhesive monomer into the partially demineralized smear layer. One study reported a higher μTBS for direct resin bonding to superfine-grit prepared dentin than to regular-grit prepared dentin when using Clearfil SE Bond, and, based on SEM findings, suggested that the greater porosity of intertubular dentin with superfine-grit preparation implied more channels of penetration for adhesive monomers²⁸⁾. This may account for the results of the present study, as the acidity of the self-etching primer in UniFil Bond is the same as that in Clearfil SE Bond ($\text{pH} = 2.0$).

On the other hand, Koase *et al.*¹²⁾ found no significant difference in μTBS between regular-grit and superfine-grit prepared dentin when using Clearfil® Protect Bond (also known as Clearfil Megabond FA in Japan), although the pH of its self-etching primer is the same as that of Clearfil SE Bond. They attributed their results to differences in monomer diffusion and the mechanical strength of the adhesive resin itself. In an earlier study, we found that tensile bond

strength in a specimen primed with SE Bond primer and bonded with Clearfil Protect Bond adhesive was significantly lower than that in a specimen primed with SE Bond primer and bonded with SE Bond adhesive⁹⁾, which supports their speculation.

With Adper Single Bond, however, no significant difference was found between regular-grit and superfine-grit prepared dentin. These results are similar to those of earlier studies on direct bonding^{21,23)}. It is worthy of note that 16 of the 28 superfine-grit prepared specimens failed before μ TBS testing, suggesting the presence of a resin-unprotected demineralized layer beneath the hybrid layer. In an earlier study, Spencer *et al.*³⁰⁾ found a 4- μ m thick layer of unprotected protein immediately under a 2- μ m thick hybrid layer when dentin was prepared with #600 silicon carbide paper and bonded with Adper Single Bond. Once the resin monomer has sufficiently penetrated the demineralized and exposed protein layer, a higher μ TBS can be expected. Conversely, insufficient penetration may result in degradation of adhesion and serious gap formation. Therefore, our null hypothesis, that bur selection would not affect microtensile bond strength between resin-coated dentin and indirect resin composite bonded with adhesive resin cement, was rejected with UniFil Bond but accepted with Adper Single Bond.

Although resin-coating with UniFil Bond for superfine-grit prepared dentin significantly contributed to an increase in μ TBS, coating with Adper Single Bond showed no such effect. One study reported a significant effect for resin coating with a UniFil Bond/Link Max combination which was not found with an Adper Single Bond/Rely XTM (3M ESPE) combination¹⁷⁾. The current results indicate that resin coating with Adper Single Bond should really be avoided, as opening of dentinal tubules at the fractured surface and the large number of pre-test failures suggests a potential route of bacterial invasion into the deep dentin and pulpal tissue. On the other hand, with regular-grit prepared dentin, resin coating showed no significant effect with either UniFil Bond or Adper Single Bond. These

results suggest that selection of both resin coat and bur are important in achieving good clinical results in indirect bonded restoration.

This study evaluated different types of two-step adhesive as resin-coating materials. Current adhesive research has focused on simplifying the application procedure to etch, prime and bond, as a one-step process. Such adhesives can be further classified by acidity as 'mild' (pH>2), 'intermediately mild' (pH = 1–2), or 'strong' (pH<1)^{14,35)}. Bond strength with superfine-grit prepared dentin and direct bonded composite with 'strong' 1-step adhesive reportedly produces a large number of failures during specimen fabrication²⁸⁾, as occurred with Adper Single Bond in the present study. In contrast, 'mild' 1-step adhesive is suggested to leave a residual smear layer at the adhesive interface. Because these mild adhesives contain a larger amount of solvents such as water, ethanol, and acetone, the mechanical strength of the polymerized adhesive itself has been reported to be weaker than that with 2-step self-etch adhesives²⁶⁾. This means that the hybridized area between the adhesive and the residual smear layer may cause defects at the adhesive interface.

Conclusion

Within the limitations of this study, the following conclusions are drawn:

1. Superfine-grit preparation of the dentin surface was affected by resin-coating with a 2-step self-etch adhesive.
2. Superfine-grit preparation of the dentin surface somewhat decreased microtensile bond strength when resin-coating with a 2-step etch & rinse adhesive was applied.
3. Selection of bur showed no effect on microtensile bond strength when no resin-coating technique was applied.

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