Gap measurement and bond strength of five selected adhesive systems bonded to tooth structure

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Abstract

The ability of a restorative material to bond and seal the interface with tooth structure is perhaps the most significant factor in determining resistance to marginal caries. Thus, the quality and durability of marginal seal and bond strength are major considerations in the selection of restorative materials. The purpose of this study was to compare the bond strength and marginal discrepancies of five adhesive systems: All-Bond 2, Clearfil Liner Bond, KB 200, ProBond and AELITE Bond. Twenty-five buccal and 25 lingual cavities were prepared in 25 caries-free extracted molar teeth, giving 10 cavities for each of the 5 adhesive systems. All teeth were restored with the resin composite Pertac Hybrid, or PRISMA Total Performance Hybrid with their appropriate adhesive systems. After restoration, the teeth were thermocycled, were stained with a 1.5% aqueous solution of a procion dye (reactive orange 14) and sectioned coronally with a saw microtome. Three sections of 200 µm thickness were prepared from each restoration which were then examined microscopically to measure marginal gap widths using a confocal tandem microscope. Shear bond strength measurements were carried out on the dentine bond using a universal testing machine. The All-Bond 2 adhesive system was found to have higher shear bond strength and to have the least gap width at the cementodentinal margin.

Key words: Bond strength, adhesive systems, marginal discrepancies.

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Introduction

The success of an adhesive bond to dentine is dependent largely on the specific state of the interface between tooth and adhesive. Most current dentine adhesive systems use pretreatment to demineralize the initial few micrometres of the dentine surface supposedly leaving a mesh work of collagen into which the adhesive resin can penetrate and polymerize. The penetration and polymerization of the adhesive is, in general, a function of the conditioner used in pretreatment procedures. Whether or not this is true, different adhesive resins have different abilities in bonding to dentine. Good adhesion between dentine and restorative resins is of primary importance in clinical practice because it should reduce marginal leakage and permit more conservative cavity preparations. Dentine bonding agents are used extensively, primarily because of the manufacturers' claims that they enhance adhesion to tooth structure. This should substantially reduce microleakage because of the development of a strong bond between tooth structure and the resin. This eliminates the need for retentive undercuts, contributes to conservative cavity preparation, and prevents the formation of gaps at the cavo-surface margins of the restorations.

Microleakage studies have demonstrated that various bonding agents differ in their ability to minimize marginal penetration of dye solutions. The newer generation adhesive systems modify the dentine to remove the smear layer or to produce a clean surface for the application of a resin bonding agent.[1] The interactions between the various components of such adhesive systems are intended to enhance micromechanical bonding to dentine.[2] Demineralization of the dentine surface is followed by penetration with an adhesion promoter thought to provide the surface energy necessary for good monomer penetration and bonding occurs when the bonding agent penetrates into the demineralized,

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Table 1. Components of materials tested

Materials	Components	Composition
All-Bond 2	Echant	10% H_3PO_4
Bisco Inc.	Primer A	2% NTG-GMA
Itasca, IL, USA	Primer B	16% BPDM
Bonding sequence: *abdegi	Resin	Bis-GMA, UDMA, HEMA
Clearfil Liner Bond	Ca Agent	10% citric acid, 20% $CaCl_2$
Kuraray-J. Morita USA Inc.	SA Primer	3% NMSA
Tustin, CA, USA	Photo Bond	10-MDP
Bonding sequence: *abcecgcij	Protect Liner	Micro-filled composite (42 m%)
AELITE Bond	Uni-etch	32% H_3PO_4
Bisco Inc., Itasca, IL, USA	Primer	Ethyl alcohol-hydroxyalkyl
Itasco IL, USA		methacrylate-diartl sulphone
Bonding sequence: *abdegi		dimethacrylate
	Bonding	Bis-GMA – Urethane dimethacrylate
		hydroxyalkyl methacrylate rocryl-700
KB-200	Primer A	Phosphoric ester adhesive monomer
Kurarary Co Ltd		(phenyl P)
Osaka, Japan	Primer B	Salicylic acid derivative monomer
Bonding *sequence:* *eghi		(5-NMSA) and hydrophilic monomer
		(HEMA)
	Bonding agent	Phosphoric ester adhesive monomer
		(MDP), hydrophilic monomer (HEMA)
		and micro-filler
ProBond	Primer	6% Penta (Dipentaerythritol)
L.D. Caulk Division		pentacrylate phosphoric acid ester)
Dentsply Int Inc		75% Acetone
Milford, DE, USA		19% Ethanol
Bonding sequence: *abdegi	Adhesive (formerly	56% UDMA resin
	Prisma Universal) Bond	36% Polymerizable monomers
		5% Penta
		2% Polymerizable initiators
		7% Glutaraldehyde

**Bonding steps:* (a) apply conditioner (or etchant); (b) rinse conditioner; (c) air dry; (d) blot dry; (e) apply primer; (f) light-cure primer; (g) apply resin; (h) thin resin with compressed air; (i) light-cure resin; (j) apply low-viscosity resin.

primed surface and is polymerized. This results in a resin-reinforced dentine layer, or hybrid layer, consisting of polymer, collagen, and hydroxyapatite.

The aims of this study were to evaluate a range of adhesive systems, to determine gap widths and bond strengths in dentine using selected composite materials, and to determine the effect of stress applied by load after thermocycling. The strength of the bond was measured as shear bond strength with a universal testing machine.‡ Marginal discrepancy and gap formation were measured at five sites on the Class V cavity margins with confocal microscopy.

Materials and methods

Caries-free molar teeth were collected immediately after extraction and were stored in 0.9% neutral saline at 4°C for a maximum of 7 days. Class V buccal and lingual cavities were prepared in each tooth to the following specifications: depth 1.5-2 mm, occluso-gingival height 2.5 mm, mesio-distally to each proximal reflection line, and a continuous 0.5 mm wide bevel was prepared in enamel at 45° to the cavo-surface margins. The cavities were prepared with a medium grit cylindrical diamond bur in a high speed handpiece with water coolant. Cavity refinement was undertaken with a steel bur and water coolant in a slow speed handpiece. Enamel bevels were placed with a flame-shaped, fine grit, cylindrical diamond bur using water spray. After a 30 second blast of air/water from a triplex syringe, each cavity was dried for 10 seconds with air from oil-free lines. An apical seal in each tooth was achieved with a glass ionomer§ restorative material.

The cavities were divided into groups, and treatments were randomly allocated. Within each tooth, one treatment was applied to a buccal cavity and a different treatment applied to the lingual surface.

‡Monsanto Hounsfield Tensometer, Test Equipment, Croydon, England.
§Ketac Silver. ESPE, Sydney, Australia.

Table 2. Combinations of adhesive and resin composite applied to the cavities

Adhesive	Composite
All-Bond 2	Pertac Hybrid (ESPE)
Clearfil Liner Bond System	Pertac Hybrid (ESPE)
KB-200	Pertac Hybrid (ESPE)
ProBond	Pertac Hybrid (ESPE)
AELITE Bond	Prisma TPH (Caulk, Dentsply)

Table 3. Composition of artificial saliva

%	Composition
0.015	Potassium chloride
0.001	Sodium fluoride
0.022	Calcium chloride $2H_2O$ BP
0.099	Disodium hydrogen phosphate anhydrous Na_2HPO_4
0.013	Magnesium acetate $4H_2O$
0.2	Hypromellose 2910 USP
1.25	Ethanol 96% BP
0.06	Methyl parahydroxyl benzoate BP
0.03	Peppermint oil BP
to 100	Deionized water

The results from the two cavities per tooth were not interrelated and the data were analysed as if they were independent replica cavities for each of the treatments. The adhesive-resin combinations were applied according to the manufacturers' instructions. The specifications for the adhesive systems applied to each group of cavities are listed in Table 1. The resins applied to groups of cavities are listed in Table 2. After complete incremental adaptation of the composite to all surfaces and margins, each slightly overfilled cavity was covered with a mylar matrix strip and the resin composite was polymerized using a visible-light unit, for 40 seconds at a distance of less than 10 mm, as per manufacturer's instructions. Excess resin composite was removed with a finishing bur and the restorations were checked for obvious marginal deficiencies with a ×10 magnification dissecting microscope. The restorations were then polished under water spray with progressively finer grades, coarse to superfine discs.¶ The teeth were painted with nail varnish to within 2 mm of the cavity margin. They were soaked overnight at room temperature (25°C) and were then thermocycled in artificial saliva (Table 3) for 500 cycles at temperatures ranging from 5°-55°C. All the teeth were stained in a 1.5% aqueous solution of a procion dye** for 60 seconds. Three occluso-gingival sections in the coronal plane, of 200 μm thickness, were taken from each cavity with a saw microtome†† and then were mounted on glass slides. Marginal gap-widths were measured using a Confocal Microscope‡‡. The gap-widths were measured as the largest visible gap at each of the five sites on these teeth as illustrated in Fig. 1.

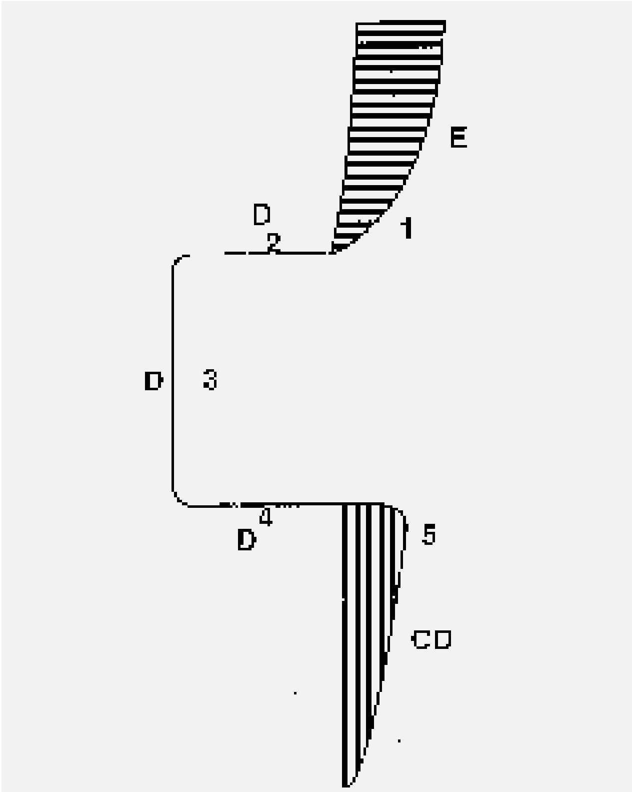

Fig. 1.–A schematic diagram of margin measurements: site 1 is the junction with the enamel bevel (E), site 2 the coronal wall in dentine (D), site 3 is pulpal wall, site 4 the radicular wall, and site 5 the gingival margin on cementum or dentine (CD).

For shear-bond strength testing, a further 25 recently extracted molar teeth were washed free of adherent blood products, and stored at 4°C in distilled water changed at 24-hour intervals. The teeth were used within two months of extraction. The tooth crowns were sectioned from the roots and flat buccal or lingual surfaces of dentine were prepared by sagittal sectioning using a diamond saw 100 μm in from the enamel surfaces. The prepared sections were then mounted in cylinders of self-curing methacrylate resin with the dentine surfaces exposed. Each exposed surface was then polished with silicon carbide abrasive paper (grade 1000, mean grit size 18 μm) under running water. Specimens were allocated on a random basis to the various experimental groups.

Specimens were stored at 25°C for 24 hours in a humidor. Prior to using the adhesives, the specimens were dried with oil-free compressed air for 15 seconds. Waterproof, 1 mm thick, double-sided, adhesive polyvinyl-chloride tape, pre-punched with 2.5 mm diameter holes, was applied to provide circular areas for the adhesion treatment.

,Demetron Research Corporation, Danbury, Connecticut, USA.
¶Sof-Lex Discs, 3M, Minnesota, USA.
**Reactive orange 14, Sigma Chemicals, USA.
††Saw microtome, Leitz 1600. Leitz, Oberkirchen, Germany.
‡‡Confocal tandem-scanning reflected microscope. Physiology Department, The University of Queensland.

Table 4. The means (M) and standard deviations (SD) of gap widths (µm) at five sites (Fig. 1) treated with five adhesives

Adhesives	Site 1		Site 2		Site 3		Site 4		Site 5	
	M	SD	M	SD	M	SD	M	SD	M	SD
All-Bond 2	1.6	1.9	1.7	1.7	3.2	4.5	4.7	5.6	2.2	2.2
Clearfil	2.5	6.7	9.3	14.3	7.5	12.9	12.3	8.8	3.7	5.6
KB 200	1.8	4.0	8.2	8.9	7.7	8.1	8.0	8.6	3.8	3.5
ProBond	0.9	1.4	7.4	4.0	5.2	3.0	4.5	3.4	5.4	2.7
AELITE Bond	0.6	1.2	3.1	2.9	7.4	13.2	11.9	10.6	8.3	4.9

The bonding agents were applied according to the manufacturers' instructions, with a fine brush via the circular windows on the treated surfaces to form a thin layer, which was then polymerized with a curing light.§§ Composite was then applied and polymerized. The shear bond strengths were then measured using a custom-made, single-plane, jig attached to a testing machine‡ fitted with an electronic load cell. The force was registered using a strain gauge (400×0.02 N). Specimens were tested to failure using a cross-head speed of 5 mm/minute. Testing was performed without knowledge of the treatment procedures undertaken. The force at failure was used to calculate shear bond strength. Means and standard deviations were calculated for each treatment, and differences between groups were analysed using a one-way, two-tailed analysis of variance. Selective post-hoc *t* tests were also undertaken. The mode of failure was determined by examination using a stereo microscope, 64× magnification,,, and was classified as either cohesive, adhesive, or mixed at each of the material interfaces according to the area of substrate free of material.

Results

Gap widths

As shown in Table 4, mean gap widths for all materials were lowest at site 1. These ranged from 0.6-2.5 µm and were widest at site 4 with a range of 4.5-12.3 µm. For most sites, All-Bond 2 had the lowest gap widths with low standard deviations. Highest gap widths were found for Clearfil and KB 200 with high standard deviations. The appearance of the gap widths as revealed by confocal tandem-scanning microscopy are illustrated in Fig. 2-4.

Concerning the gap widths achieved with enamel at site 1, the analysis of variance showed that the mean gap widths for All-Bond 2, ProBond and AELITE were not significantly different ($p>0.05$). At the wall of the cavity in dentine at site 2, the mean gap widths for all materials were significantly different from each other. On the pulpal wall, site 3, the mean gap widths for AELITE Bond and Clearfil were not significantly different, ($p>0.05$) and high standard deviations were found (Table 4). For site 4, the mean gap width for All-Bond 2 was the highest found for this material but was similar to ProBond (Table 4). The mean gap widths of AELITE Bond and Clearfil, AELITE Bond and KB 200, and also Clearfil and KB 200 were not found to be significantly different ($p>0.05$). At site 5, the margin with cementum, the mean gap width for All-Bond was still the lowest of all materials (Table 4). The gap widths of Clearfil, KB 200 and ProBond were not significantly different from one another ($p>0.05$) while AELITE had the greatest mean gap width.

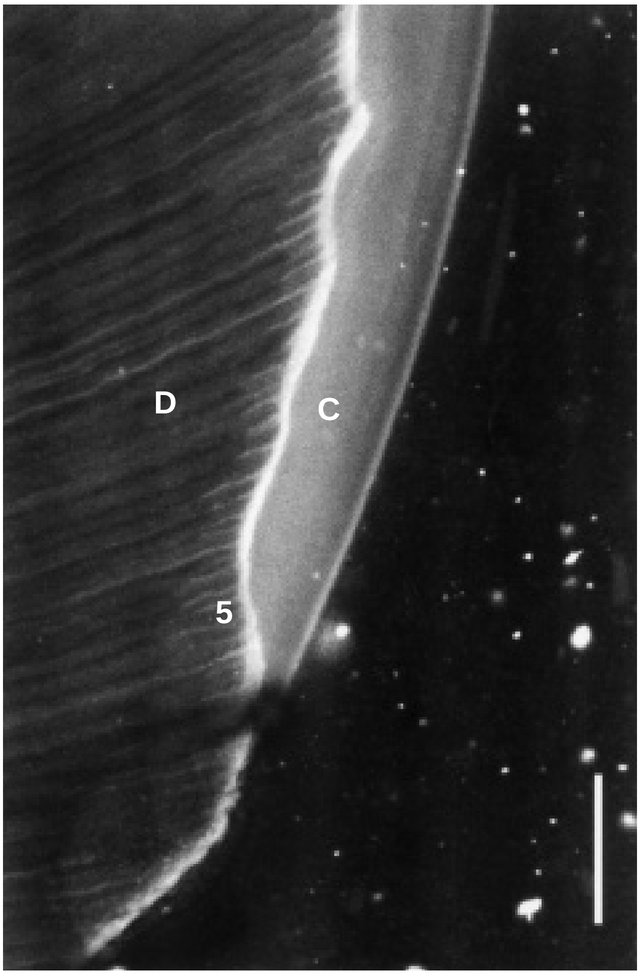

Fig. 2.–Confocal tandem-scanning image of the marginal gap between dentine (D) and resin composite (C) at site 5 with All-Bond 2 adhesive showing good marginal adaptation [Bar=50 µm].

§§Demetron Research Corporation, Danbury, CT, USA.
,,Olympus Optical Co., Tokyo, Japan.

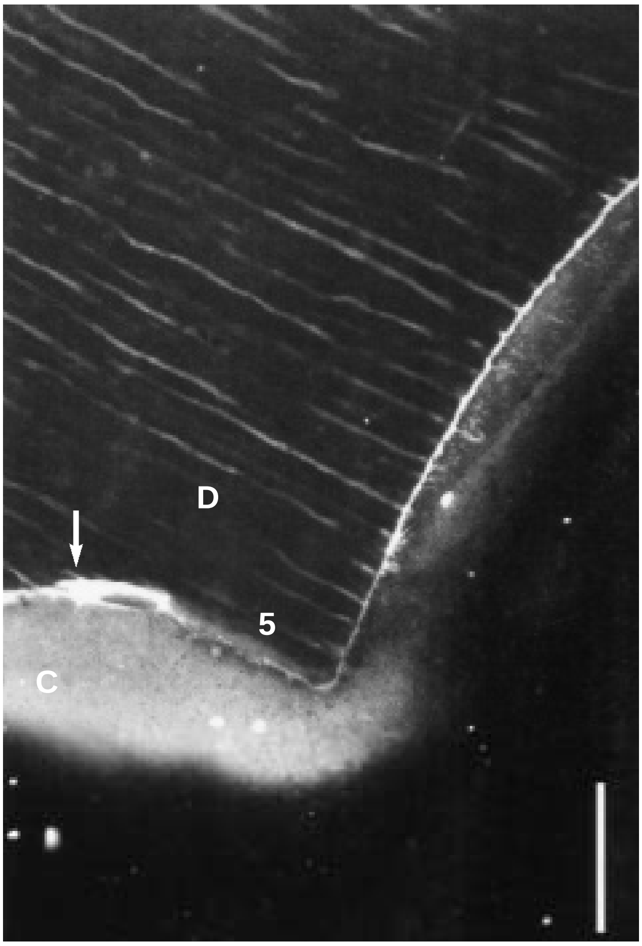

Fig. 3.–The marginal gap between dentine (D) and resin composite (C) at site 5 with Clearfil shows good adaptation but the variability (arrow) in gap width associated with this material [Bar=50 μm].

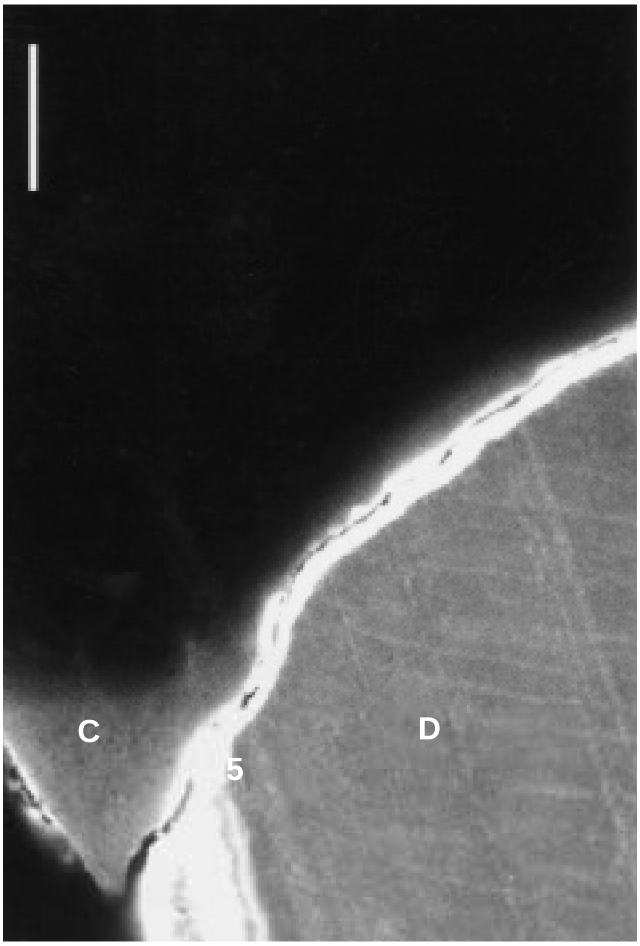

Fig. 4.–The marginal gap between dentine (D) and resin composite (C), [which was PRISMA TPM at site 5 with AELITE Bond]. Lack of dye in dentinal tubules possibly indicates good seal by AELITE. However, the gap width suggests a separation of the resin from bond by contraction [Bar=50 μm].

Shear bond strengths

Table 4 shows that the bond strengths, of the five adhesives tested, ranged from 1.17-14.8 MPa. All-Bond 2 and Clearfil Liner Bond had the highest mean bond strengths and KB 200, ProBond and AELITE Bond gave significantly lower values. The analysis of variance showed that the mean shear bond strengths for All-Bond 2 and Clearfil liner Bond were not significantly different from one another ($p>0.05$), but were significantly greater than the other materials tested. KB 200 and ProBond were also not significantly different from each other. AELITE had the lowest bond strength. The mode of bond failure was adhesive for each of the materials tested. Dentine surfaces were typically smooth and clean, indicating that fracture occurred at the interface between dentine and resin.

Discussion

The purpose of this investigation was to study the relative merits of five bonding systems to dentine and cementum. The general hypothesis tested in these experiments was that some adhesive resins are better than others in bonding to dentine and cementum. The results from the study support this hypothesis.

With respect to gap widths, All-Bond 2 performed best at all sites. The poor performance of AELITE compared with all others may have been more related to the different composite used with this bonding agent, or the resin may have had greater polymerization shrinkage (Fig. 4). All of the materials showed gap widths less than 2.5 μm at the enamel margins (site 1). This reflects the superior bond generally achieved with enamel. On the dentine walls and cavity floors, a range between 1.7 and 12.3 μm was found for sites 2, 3 and 4. This may be the result of poorer bonding to dentine or be related to polymerization shrinkage of the resin composite, with air-trapping under the composite particularly on the pulpal wall (site 3) where high standard deviations were found. All-Bond 2 had the least gap widths at the cementodentinal margin (site 5, Fig. 2).

The shear bond strength of All-Bond 2 was 14.8±5.5 MPa and of Clearfil 9.9±4.4 MPa, respectively. Two materials with bond strengths in the intermediate range were ProBond (5.8±2.1 MPa)

Table 5. The means and standard deviations of shear bond strength (MPa) of a dentine to adhesive bond

Adhesive	Mean	SD
All-Bond 2	14.8	5.5
Clearfil	9.9	4.4
KB 200	4.23	1.9
ProBond	5.84	2.1
AELITE Bond	1.17	0.6

and KB 200 (4.2±1.9 MPa) with intermediate gap widths at the cementodentinal margins. Another material (AELITE Bond), had a bond strength at the low end of the spectrum, and exhibited the highest gap widths at cemento-dentinal margins. Overall gap width appeared to correlate with shear bond strength for all the materials tested.

The standard deviations of these measurements are considered to be in agreement with other studies in this field. Fortin *et al.*[3] reported a bond strength of 9.4 MPa for All-Bond 2 and 13.3 MPa for Clearfil Liner Bond. Staninec and Kawakami[4] found equivalent shear bond strengths for Clearfil Liner Bond, which ranged from 7.8 to 9.6 MPa depending on the time of movement after bonding. Higher shear bond strengths for All-Bond 2 and for Clearfil Liner Bond have been reported. Barkmeier *et al.*[5] reported a shear bond of 39.99 MPa for All-Bond 2 whilst Perdigao *et al.*[6] reported a shear bond strength of 22.5 MPa for All-Bond 2. However, the absolute values obtained in any given study may be of less importance than the relative rank of the various systems after testing[3] particularly when correlated with gap widths.

All-Bond 2 and Clearfil Liner Bond II are newer generations of bonding agents. Both of these agents have one conditioner for both enamel and dentine to simplify handling and to shorten the application time. With the use of these materials, the dentine surface is said to be completely demineralized with the collagen fibrils being exposed and infiltrated by resin primers to a depth of up to 4 μm. This creates a resin-reinforced hybrid layer that improves the bond strength for a short period.[7] Despite its good bond strength, Clearfil II did not appear to have as good marginal adaptation generally as All-Bond 2 in these experiments. The high standard deviations of gap width for Clearfil II suggest that some properties of this adhesive are not conducive to a uniform seal.

The smaller the dentine thickness towards the pulp, the larger is the percentage of dentinal tubules exposed per unit area and in diameter. The bond strength of All-Bond 2 and Clearfil II on dentine close to the pulp may be only 30-40 per cent of the strength found in peripheral dentine.[8] The bond of currently available dentine bonding agents is regarded as a relatively weak one when compared with the bond to acid-etched enamel.[9] For some dentine adhesives, a total etch technique has been advocated as a simplified single step conditioning both the enamel and the dentine.[10] It is stated that even if the acid conditioning is confined to the enamel, rinsing of the etchant will invariably result in an acid conditioning of the dentinal walls of the cavity, with the concomitant removal of the smear layer on the cavity surface[11] and an increase in dentine permeability.[12]

Even though these experiments were carried out under laboratory conditions it is clear from these studies that the newer All-Bond 2 adhesive system should provide the best clinical results in situations where good bond strength and adaptation are required.

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