©Operative Dentistry, 2012, **37-2,** 137-149

Effect of Tooth Surface Preparation on the Bonding of Self-Etching Primer Adhesives

OA Adebayo • MF Burrow • MJ Tyas
J Palamara

Clinical Relevance

The effectiveness of some self-etching primer adhesive systems is not significantly affected by the mode of rotary instrumentation used in dentin preparation.

SUMMARY

The aim of this study was to determine the bonding effectiveness of four self-etching primer adhesives after various tooth preparation protocols. Enamel/dentin specimens were prepared from 84 permanent molars, divided into three enamel preparation groups (silicon carbide paper [SiC]; erbium, chromium:yttrium, scandium, gallium, garnet [Er,Cr:YSGG] laser [EL] and diamond bur [DB]) and five dentin preparation groups (SiC, EL, DB, steel [SB], and ceramic burs [CBs]). In each group, specimens were equally divided into four subgroups and were bonded using Clearfil SE Bond (CSE, Kuraray), Xeno IV (XE, Dentsply), Tokuyama Bond Force (TK, Tokuyama) and Filtek Silorane System Adhesive (FS, 3M ESPE), as well as a hybrid resin composite (Clearfil Majesty Esthetic, Kuraray) for CSE, XE, and TK, and Filtek Posterior Restorative (3M ESPE) for FS). After 24 hours of water storage at 37°C, microshear bond strength (μSBS) testing was carried out. Data were analyzed using analysis of variance (ANOVA)-Tukey test at α=0.05 and bond failure modes assessed. Representative debonded specimens were prepared and examined under the scanning electron microscope (SEM). All adhesives exhibited no significant differences in μSBS on enamel and dentin under the clinical cavity preparation protocols, except for TK on dentin. SEM revealed areas of altered subsurface enamel/dentin following EL ablation.

*Olabisi Asande Adebayo, BDS, Dr Med Dent, PhD, Restorative Dentistry, University of Melbourne, Melbourne, Victoria, Australia

Michael F. Burrow, BDS, MDS, PhD, MEd, MRACDS(Pros), FRACDS, Faculty of Dentistry, Hong Kong University, Hong Kong, China

Martin John Tyas, BDS, PhD, DDS, University of Melbourne, Melbourne, Australia

Joseph Palamara, BSc, PhD, Melbourne Dental School, The University of Melbourne, Carlton, Victoria, Australia

*Corresponding author: 720 Swanston Street, Carlton, Melbourne, Victoria, Australia; e-mail: adebayoo@unimelb.edu.au

DOI: 10.2341/11-172-L

INTRODUCTION

The bond strengths of enamel-dentin adhesives to enamel and dentin may be affected by various factors.[1-3] Surface characteristics of tooth structure resulting from different preparations of protocols may affect bonding effectiveness, but this may depend on the type of adhesive system used. Generally, total-etch adhesives are less affected by surface characteristics than are self-etching primer adhesives.[4-13] On enamel, surface roughness[11] while on dentin, surface roughness,[6,11,14] and smear layer quality,[5,8,15] may influence bond strengths for self-etching primer adhesives. Variability in bonding effectiveness of this group of adhesives may be attributed in part to their pH and etching aggressiveness on the enamel and dentin substrate,[16,17] and to the particular enamel-dentin adhesive used.[17-20]

Self-etching primer adhesives simultaneously demineralize and infiltrate dentin; therefore theoretically, more complete resin infiltration may be accomplished than with total-etch adhesives. Because functional and cross-linking monomers are present in a single mixture, resin infiltration to the same depth as dentin demineralization may be more likely in "all-in-one" adhesives than in their two-step counterparts. Results of a previous study by the present authors, in which four recent "all-in-one" adhesives exhibited no significant differences in microshear bond strength, irrespective of dentin tubule orientation and depth,[21] in contrast to two-step self-etching primer adhesives, appear to support this.

Tooth preparation is accomplished with the aid of hand and/or powered cutting instruments, including rotary and laser instruments. Conventional cavity preparation using rotary instruments usually involves the use of more than one type of such instruments. It is important to ascertain the effects on enamel and dentin bond strength of various surface characteristics that may result from such cavity preparation.

With the advent of "minimal intervention" dentistry, more conservative modes of tooth preparation have been introduced. The use of erbium lasers for enamel and dentin preparation has been proposed. However, conflicting reports have described the effectiveness of resin bonding following laser tooth preparation.[11,22-27] Increased resistance to acid etching of Er:YAG-irradiated enamel has been reported.[28] Other authors have reported a significant increase in calcium and phosphate concentrations in irradiated dentin at the cavity floor following erbium, chromium:yttrium, scandium, gallium, garnet (Er,Cr:YSSG) irradiation.[29] Another study reported a significant increase in quantities of calcium in Er,Cr:YSSG-irradiated canine mandibular bone, although the calcium:phosphate ratio was not significantly affected.[30] A more mineralized, acid-resistant enamel surface may resist etching by the weak acid of a self-etching primer adhesive.[17] A more mineralized enamel surface, such as could arise following laser irradiation, may result in poorer etching and lower bond strengths.

More recently, a novel innovation for the excavation of soft, carious dentin,[31] a ceramic bur (KISM-Cera Bur, Komet, Lemgo, Germany) was developed. There are as yet no reports on the effect on cavity preparation of using the ceramic bur on resin bonding.

A resin composite based on a new technology using silorane resin was recently introduced. The name "silorane" is derived from its chemical building blocks of siloxanes and oxiranes.[31] The combination of the properties of siloxanes and oxiranes results in a resin composite that the manufacturer claims is a biocompatible, hydrophobic, low-shrinkage product.[31] The novel resin matrix requires a specific two-part self-etching primer adhesive. Although the primer component of the adhesive is made up of hydrophilic methacrylate–based resins similar to those of other adhesive systems,[31] the hydrophobic adhesive bond, developed to be compatible with the new silorane restorative resin,[31] has been reported to exhibit lower polymerization stress and shrinkage. The microshear bond strengths exhibited by this adhesive to enamel and dentin after different tooth preparation conditions are not known.

The aim of this study was to determine the microshear bond strengths of one two-step self-etching primer adhesive, two "all-in-one" adhesive systems, and the silorane-based adhesive to enamel and dentin, prepared using a high-speed flat-fissure medium-grit diamond bur, a slow-speed cross-cut flat-fissure steel bur, a round ceramic bur, and an Er,Cr:YSGG laser. The null hypothesis tested was that there is no difference in microshear bond strengths of one two-step self-etching primer adhesive, two "all-in-one" adhesives, and the silorane-based adhesive to enamel and dentin prepared using various tooth preparation methods.

MATERIALS AND METHODS

Ethics approval was obtained from the University of Melbourne Human Research Ethics Committee for

the collection and use of 84 whole human permanent molar teeth from the Royal Dental Hospital in Melbourne. The teeth were stored in 1% chloramine T (pH = 9.1) solution for two weeks, transferred into phosphate-buffered saline solution (pH = 7.2) at 4°C, and used within six months of extraction. Twenty-four teeth were used for enamel specimen preparation and were sectioned at the cementoenamel junction and perpendicular to the occlusal surfaces mesiodistally and buccolingually to obtain 96 enamel specimens. Sixty-four enamel specimens from 16 teeth were mounted in dental stone in plastics molds with the enamel surface exposed, labeled according to tooth number and ground with 600-grit silicon carbide paper (SiC). These specimens were divided into two groups for tooth preparation using 600-grit SiC paper as control and the erbium laser (EL, Waterlase; Biolase Technology Inc, San Clemente, CA, USA); each group comprised 32 specimens from eight teeth. The 32 enamel specimens from the remaining eight teeth were mounted as above but were not ground with SiC paper. These constituted the specimens that were prepared with a high-speed medium-grit flat-fissure diamond bur (DB, average particle size 100 μm, DB 835 314 012). Surface enamel was abraded by the diamond bur in a hand-held high-speed handpiece (Trend, TC 95BC, W & H, Bürmoose, Austria) with two straight strokes to obtain a flat surface. The bur was changed after every four preparations.

The erbium laser was used with the following characteristics: wavelength 2780 nm, power output range of 0–6 W, pulse duration 140 μs, repetition rate 20 Hz, and pulse energy of 0–300 mJ. A G6 fiberoptic sapphire tip, 6 mm in length and with a spot size of 600 μm diameter, was used in a noncontact, focused mode held perpendicular and 1–2 mm away from the surface being ablated. The enamel was irradiated at a power setting of 5.5 W (energy density 171.9 J/cm^2) with air pressure 90% and water pressure 80%, and was moved back and forth until the whole surface was ablated. An average enamel surface area of 4 mm × 4 mm was irradiated for five seconds. The 32 enamel specimens from each tooth preparation group were further divided into four subgroups comprising the eight specimens from two teeth for bonding with one of the two-step self-etching primer adhesives, Clearfil SE Bond (CSE, Kuraray Medical, Okayama, Japan); two "all-in-one" adhesives—Xeno IV (XE, Denstply Caulk, Milford, DE, USA) and Tokuyama Bond Force (TK, Tokuyama Dental Corp, Tokyo, Japan); and the Filtek Silorane Adhesive System (FS, 3M ESPE, St Paul, MN, USA). Details of the materials are provided in Table 1. After tooth preparation, the enamel was dried, enamel/dentin adhesives were applied according to manufacturers' instructions (Table 2), and three to four 0.75-mm diameter and 1.5-mm high translucent polyvinylchloride microtubes were placed on the adhesive surface before curing and the adhesive light-cured using a light-emitting diode light unit with an output intensity of 800 mW/cm^2 (Bluephase C8, Ivoclar Vivadent, AG, Schaan, Liechtenstein). The intensity of the curing light was checked before use. A hybrid resin composite (Clearfil Majesty Esthetic, Kuraray Medical) was loaded into the tubes and cured for 20 seconds. For specimens bonded with FS, the Filtek Silorane Posterior Restorative (3M ESPE) was loaded into the microtubes and cured for 40 seconds.

The remaining 60 molar teeth were used in dentin specimen preparation. The occlusal thirds of the crowns were removed by sectioning perpendicular to the tooth long axes, the exposed dentin surfaces checked to confirm complete removal of the enamel, and the crowns sectioned at the cementoenamel junctions. The dentin discs were mounted in dental stone and wet-ground with 600-grit SiC paper. Dentin specimens were divided into five groups of 12 specimens each for tooth preparation using the DB, a slow-speed cross-cut flat-fissure steel bur (SB, S36204 014, Komet), a slow-speed round ceramic bur (CB, K4547 014, Komet), EL, and SiC paper as controls. The high-speed handpiece carrying the DB was run across the dentin surface in four straight strokes until the whole surface was cut. The SB and CB were used in a slow-speed handpiece (WD-75, W & H) and moved back and forth on the surface until the whole dentin surface was cut. Dentin was lased using the erbium laser at a power setting of 3.5 W (energy density 109.4 J/cm^2), an air pressure setting of 65%, and a water pressure of 60%. An average dentin surface area of 8 mm × 6 mm was irradiated for 15 seconds. On completion of the tooth preparations, the 12 dentin specimens in each preparation group were further divided into four groups of three for bonding with the four dentin adhesives (Table 2). Resin bonding was carried out as described for the enamel specimens; however, six to eight microtubes were bonded per dentin disc.

The specimens were placed in distilled water in an incubator at 37°C for 24 hours. Plastics molds with the embedded specimens were mounted in a jig with the enamel/dentin surfaces flush with the external surface of the jig. A wire loop (0.35-mm diameter) was wound around the bonded cylinder with the

Table 1: *Materials*

Adhesive	Code	pH	Contents	Manufacturer	Batch No.
Clearfil SE Bond	CSE	2.0	Primer: 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), 2-hydroxyethyl methacrylate (HEMA), hydrophilic dimethacrylate, di-camphoroquinone, *N,N*-diethanol-*p*-toluidine, water Bond: 10-MDP, Bisphenol A diglycidylmethacrylate (Bis-GMA), HEMA, hydrophobic dimethacrylate, di-camphoroquinone, *N,N*-diethanol-*p*-toluidine, silanated colloidal silica	Kuraray Medical, Okayama, Japan	51766
Xeno IV	XE	~2.1	Mono-, di-, and trimethacrylate resins, dipentaerythritol penta acrylate monophosphate, cetylamine hydrofluoride, acetone, water	Dentsply-Caulk, Milford, DE, USA	080411
Tokuyama Bond Force	TK	2.3	Methacryloyloxyalkyl acid phosphate, C2-4 alkyl, HEMA, Bis-GMA, triethylene glycol dimethacrylate (TEGDMA), camphoroquinone, purified water, alcohol	Tokuyama Dental, Tokyo, Japan	YT11407
Clearfil	—	—	Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, silanated barium glass filler, prepolymerized organic filler, di-camphoroquinone	Kuraray Medical, Okayama, Japan	00003C
Majesty Esthetic					00004D
Filtek Silorane System Adhesive	FS	2.7	Primer: HEMA, Bis-GMA, phosphoric acid methacryloxy-hexylesters, 1,6-hexanediol dimethacrylate, copolymer of acrylic and itaconic acids, (dimethylamino)ethyl methacrylate, di-camphoroquinone, phosphine oxide, silane-treated silica, water, ethanol Bond: substituted dimethacrylate, TEGDMA, phosphoric acid methacryloxy-hexylesters, 1,6-hexanediol dimethacrylate, di-camphoroquinone, silane-treated silica	3M ESPE, St Paul, MN, USA	20080311
Filtek Silorane Posterior Restorative	—	—	3,4-epoxycyclohexylcyclopolymethylsiloxane, bis-3,4-epoxycyclohexylethyl-phenyl-methylsilane, mixtures of resin and siloxane by-products, silane-treated quartz, yttrium trifluoride	3M ESPE, St Paul, MN, USA	20080311

composite-dentin interface at one end and was attached to a load cell connected to a computer at the other end. Microshear bond strength testing was carried out on a universal testing machine (Imperial 1000, Mecmesin, West Sussex, UK) using the corresponding computer software (Emperor, version 1, Mecmesin) at a cross-head speed of 1 mm/min until failure occurred. Maximum loads at failure were recorded and converted to MPa by dividing the failure load by the bonded specimen surface area. Logarithmic transformation of the data was done to satisfy the assumptions of the model for statistical analyses. Means and standard deviations were obtained for each adhesive for the tooth preparation methods. Random effects mixed model analysis of variance (ANOVA) was used with the compound symmetry covariance matrix option for analyses of data obtained from samples from the same tooth. Bonferroni correction was used to compare preparation methods. Statistical analyses were carried out using the Statistical Package for the Social Sciences (SPSS), version 17 software for Windows (SPSS Inc, Chicago, IL, USA), at a significance level of $p<0.05$. Bond failure modes were assessed using a light microscope at 100× magnification and were classified as follows:

Table 2: *Bonding Procedures*[a]

Adhesive	Priming	Bonding	Light Cure	Resin Composite/ Light Cure
Clearfil SE Bond	Primer applied, left undisturbed for 20 s, air-thinned for 5 s	Bond applied and gently air-blown for 3 s	10 s	Clearfil Majesty Esthetic/ 20 s
Xeno IV	Adhesive applied with a scrubbing action for 15 s, reapplied in same manner for 15 s, gently air-thinned for 5 s		10 s	Clearfil Majesty Esthetic/ 20 s
Tokuyama Bond Force	Adhesive applied, left in place for 20 s, air-thinned for 3 s		10 s	Clearfil Majesty Esthetic/ 20 s
Filtek Silorane System Adhesive	Primer applied, rubbed in for 15 s, air-thinned for 3 s, light-cured for 10 s	Bond applied, gently air-thinned for 5 s	10 s	Filtek Silorane Posterior Restorative/40 s

[a] *Light-curing was carried out using a light-emitting diode light unit with intensity of 800 mW/cm^2 (Bluephase C8, Ivoclar Vivadent, AG, Schaan, Liechtenstein).*

- A = Adhesive bond failure, involving more than 50% of the bonded surface
- C = Cohesive failure in resin composite, involving more than 50% of the bonded surface
- M = Mixed bond failure involving up to 50% each of adhesive and cohesive failures

Debonded specimens representative of the adhesives and tooth preparation methods were retrieved from their molds, cleaned ultrasonically, embedded in epoxy resin (Epofix, Struers, Copenhagen, Denmark) for 24 hours, and sectioned perpendicular to the bonded surfaces. Exposed cross-sectional surfaces were polished with 600, 1200, 2000, and 4000-grit SiC papers, then 3 μm, 1 μm, and 0.25 μm diamond pastes; they were ultrasonically cleaned for 30 minutes, etched with 10% orthophosphoric acid for three to five seconds, rinsed for five seconds, placed in 5% NaOCl solution for five minutes, and rinsed under running water for five minutes. Specimens were dehydrated in an ascending ethanol:water series (10%, 30%, 50%, 70%, 90%, 100%) for one hour in each with at least three changes in 100% ethanol and critical point-dried (ethanol/CO_2) (CPD 030, Bal-Tec AG, Balzers, Liechtenstein). Immediately after drying, the specimens were mounted on aluminum stubs, gold sputter–coated, and examined under the field emission scanning electron microscope (FE-SEM, Quanta 200F, FEI, Hillsboro, OR, USA). Two enamel and dentin specimens were prepared using each of the tooth preparation methods.

RESULTS

Statistical analyses using random effects mixed model ANOVA showed that microshear bond strengths varied according to tooth preparation on enamel (p=0.002) and dentin (p=0.002), and also varied according to enamel-dentin adhesive on enamel (p=0.001). The results of mixed model ANOVA and Bonferroni correction for microshear bond strengths are shown in Tables 3 and 4. Results of analyses showed that bond strengths to enamel (Table 3) were significantly different between SiC and EL only for CSE (p=0.29) and XE (p=0.36). For TK, bond strengths to enamel were not significantly different between groups.

For dentin (Table 3), the microshear bond strengths of CSE and XE were not significantly affected by tooth preparation methods. Bond strengths for TK were significantly lower ($p<0.05$) following EL in comparison with other preparation methods.

At least 62% of bond failures in each enamel adhesive/tooth preparation group were mixed failures, followed by adhesive failures and a few cohesive failures (Figure 1). On dentin, bond failures in each adhesive/tooth preparation were also mainly mixed in nature (70%), except after bonding with XE on dentin prepared using the cross-cut flat-fissure steel bur, where bond failure was completely adhesive (Figure 2). Adhesive failures were also observed in the other adhesive/tooth preparation groups, but no cohesive failures were observed in dentin.

Table 3: *Microshear Bond Strength Test Results**†*

Tooth Preparation Protocol	Clearfil SE Bond	Xeno IV	Tokuyama Bond Force
Enamel (n=24)			
600-grit SiC paper	22.0 (4.48)^{A}	14.6 (3.43)^{A}	12.1 (3.40)^{A}
Medium (100-μm)-grit diamond bur	18.9 (5.12)^{A,B}	11.6 (2.93)^{A,B}	10.2 (4.44)^{A}
Er,Cr:YSGG laser	12.5 (2.80)^{B}	10.1 (3.16)^{B}	14.3 (4.19)^{A}
dentin (n=20)			
600-grit SiC paper	14.7 (4.85)^{A}	13.0 (4.79)^{A}	11.8 (3.21)^{A}
Medium (100-μm)-grit diamond bur	13.0 (5.05)^{A}	12.2 (3.95)^{A}	12.6 (2.96)^{A}
Steel bur	10.3 (4.58)^{A}	12.6 (5.73)^{A}	11.6 (4.47)^{A}
Ceramic bur	11.8 (5.37)^{A}	11.3 (4.82)^{A}	12.1 (3.41)^{A}
Er,Cr:YSGG laser	9.00 (3.35)^{A}	10.4 (2.85)^{A}	6.78 (2.12)^{B}

** Means (SD) in MPa.*
† Statistical analyses were carried out using random effects mixed model ANOVA and Bonferroni test at $\alpha=0.05$. Logarithmic transformation of microshear bond strength data was used in the analyses to satisfy the assumption of the model. Within the same columns, values with different superscript letters are significantly different for either substrate.

For FS (Table 4), microshear bond strengths to enamel varied according to enamel preparation ($p<0.001$). Bond strength of FS was significantly different between SiC and EL ($p=0.005$). On dentin, bond strengths between tooth preparation groups were not significantly different. Similar to observations made with the other three self-etching primer adhesives, bond failure modes for FS (Figure 3) on enamel were mainly mixed (85%), followed by a few adhesive and cohesive failures. On dentin, mixed failures constituted 70%, followed by adhesive failures. No cohesive failures were observed.

FE-SEM images of the cross-sectional surfaces of debonded specimens showed a remarkable difference between enamel and dentin prepared with SiC paper and the erbium laser. The typical keyhole appearance of enamel prisms was not observed in the enamel immediately underlying the lased surface (Figure 4a,b); instead there appeared to be collapse of the enamel prismatic structure. Subsurface crack formation with infiltration of adhesive resin into the cracks was observed within and underlying the lased enamel. This zone of altered enamel appeared distinctively different from the sound enamel below it. On dentin, cross-sectional views of the debonded surface revealed that the dentin immediately underlying the lased surface appeared structurally different from the sound dentin below it and from dentin prepared with SiC paper (Figure 5a,b). An area of altered dentin was observed that exhibited fewer resin tags and appeared denser in consistency, with collagen fibrils less readily visible. A layer appeared to be demarcating altered dentin from the sound dentin below it.

DISCUSSION

Reports have described the variability of bond strengths exhibited by self-etching primer adhesives under different tooth preparation protocols.[4-8,11,13,14,23,32] On enamel, conflicting reports have been put forth on the efficacy of laser preparations and the effectiveness of subsequent resin bonding. Some reports have found no significant difference in microtensile bond strengths of a two-step self-etching primer adhesive vs Er:YAG-lased enamel.[11,23] Other reports found significant lowering of enamel bond strengths of the same two-step adhesive, a one-step self-etching primer adhesive, and a total-etch adhesive following Er,Cr:YSGG[33] and Er:YAG laser ablation,[11] in comparison with a

Table 4: *Microshear Bond Strength Results for Filtek Posterior Restorative System**†*

Tooth Preparation Protocol	Filtek Posterior Restorative System
Enamel (n=20)	
600-grit SiC paper	17.1 (3.21)[a]
Medium (100μm)-grit diamond bur	10.2 (2.16)[a,b]
Er,Cr:YSGG laser	8.94 (1.75)[b]
Dentin (n=20)	
600-grit SiC paper	6.37 (1.52)[a]
Medium (100μm)-grit diamond bur	7.90 (2.14)[a]
Steel bur	8.23 (2.59)[a]
Ceramic bur	7.17 (2.09)[a]
Er,Cr:YSGG laser	8.02 (1.53)[a]

** Means (SD) in MPa.*
† Statistical analyses were carried out using random effects mixed model ANOVA and Bonferroni test at $\alpha=0.05$. Logarithmic transformation of microshear bond strength data was used in the analyses to satisfy the assumption of the model. Within the same columns, values with different superscript letters are significantly different for either substrate.

medium-grit diamond bur. In yet another study, the bond strength of one "all-in-one" adhesive was not different between Er,Cr:YSGG-lased and medium-grit diamond bur–cut enamel.[33]

Our study revealed no significant differences in bond strength on diamond bur–cut enamel and lased enamel for the two-step self-etching primer adhesive and two "all-in-one" adhesives. This result is similar in part to those of Cardoso and others,[33] who reported no change in microshear bond strength for the "all-in-one" adhesive used in their study but in part contrasts with reported significant lowering of bond strength of the two-step adhesive to enamel following Er,Cr:YSGG laser ablation compared with diamond bur–cut enamel. Kim and others[28] reported increased resistance to acid etching of Er:YAG-irradiated enamel. Hossain and others[29] reported a significant increase in calcium and phosphate concentrations in irradiated dentin at the cavity floor following Er,Cr:YSSG irradiation. Furthermore, Kimura and others[30] reported a significant increase in quantities of calcium in Er,Cr:YSSG-irradiated canine mandibular bone, although the calcium:phosphate ratio was not significantly affected. A more mineralized, acid-resistant enamel surface may resist etching by the weak acid of a self-etching primer adhesive.[17] A more mineralized enamel surface, such as could arise following laser irradiation, may thus result in poorer etching and could explain in part the lower bond strengths observed for the two-step adhesive following laser irradiation. A recent finding by the same authors[34] of a weak negative correlation between enamel microhardness and the bond strength of the two-step self-etching primer adhesive used in this study appears to support this argument.

Silicon carbide paper, usually a 600-grit surface, is used in the laboratory to prepare specimens, simulating clinical dentin/enamel preparation using a medium-grit diamond bur. Our study found no significant difference in bond strengths between the two methods on enamel and dentin for all adhesives; however, higher bond strengths were recorded with SiC. This suggests that laboratory microshear bond strengths using SiC may be higher than what may be obtainable under clinical tooth preparation conditions if a medium-grit diamond bur was used. However, significantly lower bond strengths were noted on lased enamel compared with SiC for three of the adhesives. This outcome may be explained by SEM findings of microcracks and structural alterations in enamel, which could have compromised resin bonding. SEMs of the subsurfaces of debonded lased enamel (Figure 4) revealed areas of altered enamel in which the typical keyhole appearance of enamel prisms was not present; instead, there appeared to have been collapse, cracking, and shattering of the enamel prismatic structure. The Er,Cr:YSGG laser is a hydrokinetic system; during irradiation, water ejected from an air-water spray onto the tooth surface is absorbed by incident radiation, causing heating and water evaporation, which results in high-stream pressure, which in turn induces microexpansion and explosion of dental hard tissues.[33] The Er,Cr:YSGG laser has been reported to result in enamel melting and recrystallization[33] and subsurface grooving.[35] Such a surface may compromise resin penetration and/or introduce weaknesses in the bond, which may lead to premature bond failure and low bond strengths.

On dentin, the adhesives exhibited no statistically significant difference in microshear bond strength after various tooth preparation protocols, except for

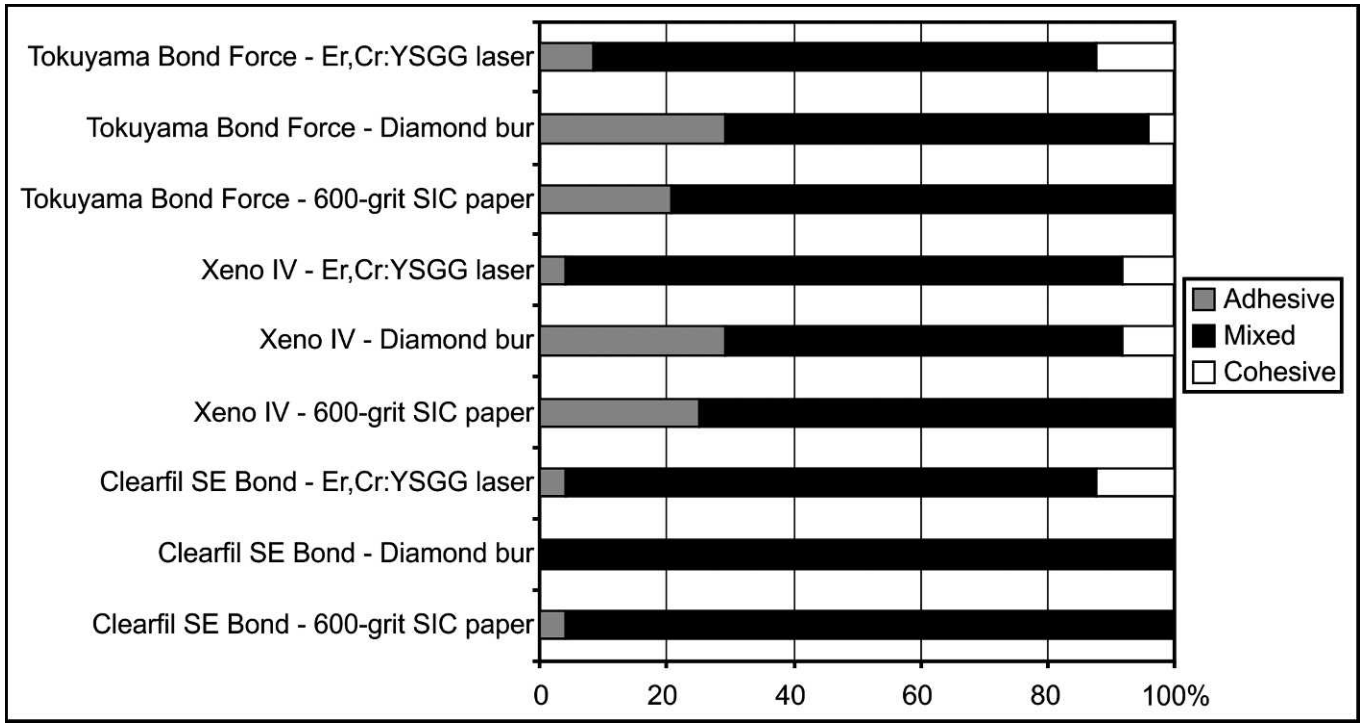

Figure 1. *Bond failure modes observed on enamel. Adhesive bond failure—involving more than 50% of the bonded surface; cohesive failure in resin composite—involving more than 50% of the bonded surface; and mixed bond failure—involving up to 50% each of adhesive and cohesive failures.*

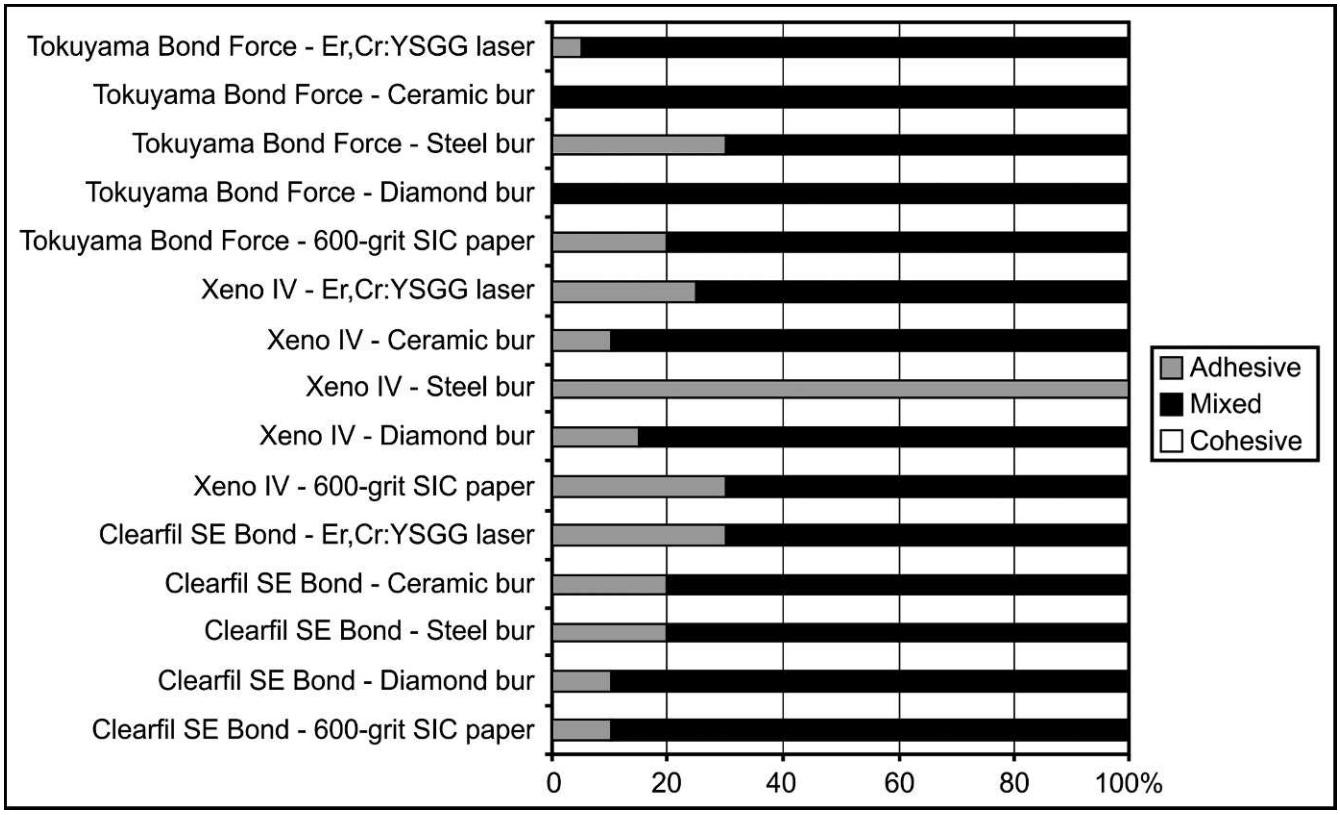

Figure 2. *Bond failure modes observed on dentine. Adhesive bond failure—involving more than 50% of the bonded surface; cohesive failure in resin composite—involving more than 50% of the bonded surface; and mixed bond failure—involving up to 50% each of adhesive and cohesive failures.*

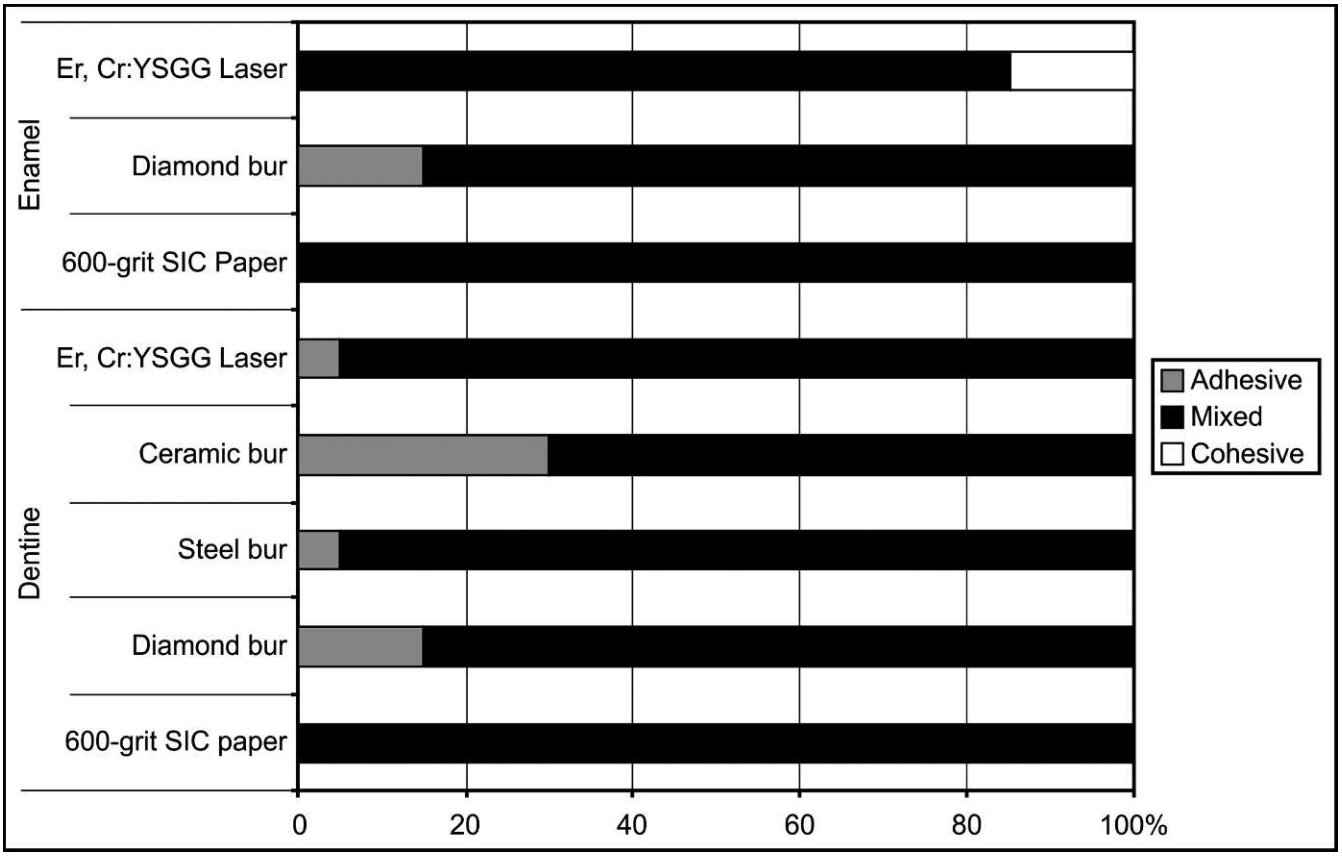

Figure 3. *Bond failure modes observed for the Filtek Silorane Posterior Restorative. Adhesive bond failure—involving more than 50% of the bonded surface; cohesive failure in resin composite—involving more than 50% of the bonded surface; and mixed bond failure—involving up to 50% each of adhesive and cohesive failures.*

one "all-in-one" adhesive, which reported lower bond strengths following Er,Cr:YSGG laser ablation.

Results suggest that bond strengths of the self-etching primer adhesives used in this study to dentin may not be affected by the type of rotary instrument used. However, variability in bond strengths of two-step self-etching primer adhesives in relation to "all-in-one" adhesives to dentin surface preparation has been reported in previous studies.[10,14,36] Semeraro and others[10] reported that two "all-in-one" adhesive systems exhibited no significant differences in microtensile bond strength to dentin prepared with regular and superfine-grit diamond burs. In the same study,[10] another "all-in-one" adhesive and a two-step self-etching primer adhesive exhibited significantly higher bond strengths with a finer surface finish. In another study, however, Ermis and others[37] reported no significant difference in microtensile bond strength of the two-step self-etching primer adhesive and one "all-in-one" adhesive bonded to dentin prepared with medium-, fine-, and extra-fine–grit diamond burs. However, lower bond strengths were observed for another "all-in-one" adhesive used.[37] In yet another study, Inoue and others[14] reported significantly higher bond strength of a one-step (two-bottle) self-etching primer adhesive to dentin finished with a superfine-grit diamond bur compared with a regular-grit diamond bur. The variable bond strength results obtained may be explained by factors such as the thickness of the dentin smear layer after preparation,[8,15] the aggressiveness of the adhesive primers,[20] and the nature of the dentin surface, whether sound or sclerotic.[38]

In contrast to the results obtained in our study, Ogata and others[13] reported significantly lower bond strength of three two-step self-etching primer adhesives to dentin prepared with a cross-cut flat-fissure steel bur compared with a medium-grit diamond bur. The difference in results may lie in the quality of the smear layer produced following dentin preparation. In the above study, the authors reported that steel burs at a speed of 2000 rpm were used; those used in our study had a speed of up to 40,000 rpm. This

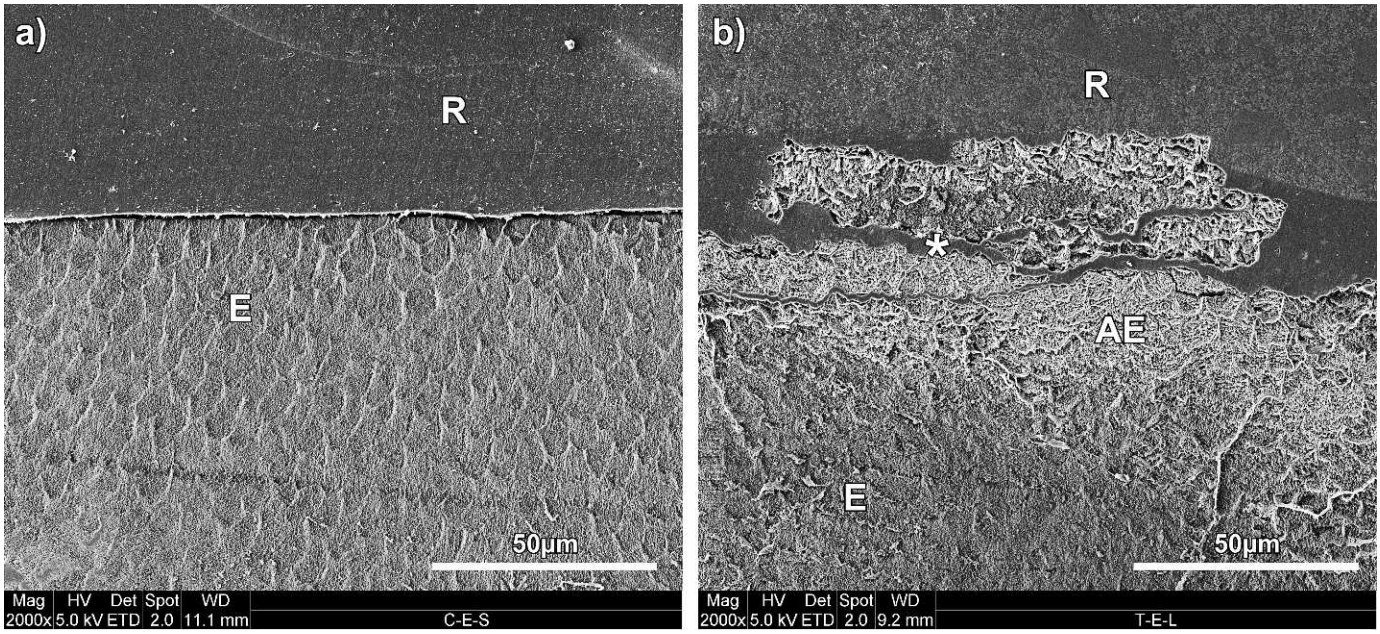

Figure 4. *Representative FE-SEM images of cross-section of a) enamel surface prepared with 600-grit silicon carbide paper; and b) enamel prepared with the Er,Cr:YSGG laser. The typical keyhole appearance of enamel prisms (as seen in part a) is distorted in the enamel immediately beneath the lased surface. Collapse of the enamel prismatic structure and subsurface crack formation within the altered enamel are evident. Resin infiltration into the cracks has occurred in the underlying enamel as the result of crack formation (asterix). AE, altered enamel; E, sound enamel; R, resin.*

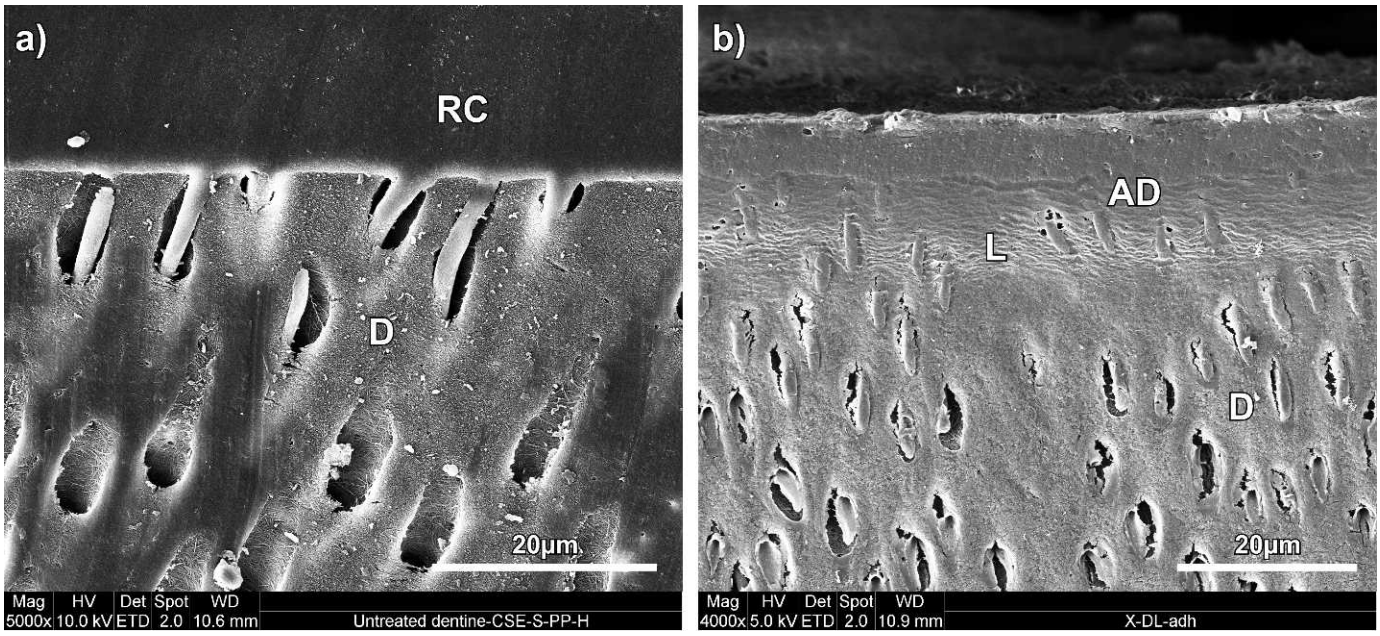

Figure 5. *Representative FE-SEM of a) bonded interface of dentin prepared with 600-grit silicon carbide paper; and b) cross-section of debonded dentin surface prepared with Er,Cr:YSGG laser. The dentin immediately beneath the debonded surface (AD) appears to be different in structure to the sound dentin further below (D). This altered dentin appears to be denser in consistency and exhibits fewer resin tags, and a layer (L) appears to be demarcating the altered dentin from the sound dentin. AD, altered dentine; D, sound dentine; RC, resin composite.*

difference in speed of cut may produce different stresses on dentin and different smear layer characteristics, which may in turn influence bond strengths. The novel ceramic bur resulted in comparable dentin microshear bond strengths to other tooth preparation protocols and therefore appears promising. Failure modes were mainly mixed in nature and consistent with other studies.[21, 37]

The lowest bond strengths were reported following dentin laser ablation for the two-step and both "all-in-one" adhesives, but this was significant only for TK. Other changes brought about by laser tooth preparation that could weaken adhesion include formation of microcracks beneath the hybrid layer, collagen denaturation due to selective ablation of organic tissue resulting in less collagen being exposed,[23, 29] and deficient dentin hybridization.[36] Martinez-Insua and others[35] also reported the presence of widespread subsurface grooving in Er:YAG-lased enamel and dentin. Crack formation and other alterations observed in subsurface enamel and dentin in this study could be attributed to thermal changes caused by laser irradiation. SEM evaluation of the subsurface of the debonded lased dentin (Figure 5) revealed dentin that appeared more dense in consistency, exhibited fewer resin tags, and appeared demarcated from the sound dentin below it by a distinctive layer. These changes observed in the lased enamel could have weakened the dental tissue and impeded resin infiltration and dentin hybridization, predisposing to premature bond failure and thus significantly lower bond strength observed for the "all-in-one" adhesive. With the altered state of the dentin, the viscosity of the adhesive and its mode of application (i.e., with or without scrubbing) may play a more important role in assisting adhesive penetration into dentin, and this may have had an effect on the bond strength outcome on dentin.

The silorane-based adhesive exhibited no significant difference in microshear bond strength to enamel and dentin under the clinical tooth preparation protocols. Low microshear bond strengths were, however, observed with the adhesive on dentin. This newly developed resin composite is hydrophobic, and its low polymerization shrinkage is attributed to the cationic ring-opening reaction, which results in a gain in chain length and subsequent lower polymerization contraction compared with the radical addition polymerization of methacrylate resins.[39] The pH of the silorane-based self-etching primer[39] is the least acidic of all the adhesives used in this study. The demineralizing aggressiveness on dentin of a self-etching primer adhesive has also been reported to be related to its pH.[20] The adhesive primer is cured before application of the bond; dentin hybridization may thus be entirely dependent on the degree of demineralization, penetration, and cross-linking produced by the primer. The pH and the resultant hydrophilicity of the silorane adhesive primer may greatly determine the extent of resin permeation into dentin. Additional studies are required.

Findings in this study show that the microshear bond strengths of the two-step self-etching primer adhesive, one "all-in-one" adhesive, and the Filtek Silorane Posterior Restorative System were not affected by tooth preparation methods. However, the bond strength of one "all-in-one" adhesive was significantly lower after laser ablation. Therefore, the null hypothesis cannot be accepted.

CONCLUSIONS

Although the microshear bond strength of one "all-in-one" adhesive may be significantly affected by dentin laser ablation, the bond strengths of all other self-etching primer adhesives used in this study, including the silorane adhesive, were not significantly affected by tooth preparation methods. Alterations in subsurface enamel and dentin, which may compromise resin bonding, were observed following Er,Cr:YSGG laser ablation.

Acknowledgements

This study was supported by an educational research grant from Dentsply Australia. The authors also acknowledge the generous donation of materials provided by the following companies: Kuraray Medical, Okayama, Japan; Tokuyama Dental, Tokyo, Japan; Dentsply-Caulk, Milford, DE, and 3M ESPE, St Paul, MN; and the Er,Cr:YSGG laser (Waterlase) by Biolase Technology Inc, San Clemente, CA.

(Accepted 29 July 2011)

REFERENCES

1. Adebayo OA, Burrow MF, & Tyas MJ (2008) Bond strength test: Role of operator skill *Australian Dental Journal* **53(2)** 145-150.
2. Al-Salehi SK, & Burke FJ (1997) Methods used in dentin bonding tests: An analysis of 50 investigations on bond strength *Quintessence International* **28(11)** 717-723.
3. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, & Carvalho RM (1995) Adhesion testing of dentin bonding agents: A review *Dental Materials* **11(2)** 117-125.
4. Hosoya Y, Shinkawa H, Suefiji C, Nozaka K, & Garcia-Godoy F (2004) Effects of diamond bur particle size on dentin bond strength *American Journal of Dentistry* **17(5)** 359-364.
5. Koibuchi H, Yasuda N, & Nakabayashi N (2001) Bonding to dentin with a self-etching primer: The effect of smear layers *Dental Materials* **17(2)** 122-126.
6. Nikaido T, Yamada T, Koh Y, Burrow MF, & Takatsu T (1995) Effect of air-powder polishing on adhesion of bonding systems to tooth substrates *Dental Materials* **11(4)** 258-264.
7. Ogata M, Harada N, Yamaguchi S, Nakajima M, & Tagami J (2002) Effect of self-etching primer vs phosphoric acid etchant on bonding to bur-prepared dentin *Operative Dentistry* **27(5)** 447-454.

8. Oliveira SSA, Pugach MK, Hilton JF, Watanabe LG, Marshall SJ, & Marshall GW (2003) The influence of the dentin smear layer on adhesion: A self-etching primer vs. a total-etch system *Dental Materials* **19(8)** 758-767.

9. Sattabanasuk V, Vachiramon V, Qian F, & Armstrong SR (2007) Resin-dentin bond strength as related to different surface preparation methods *Journal of Dentistry* **35(6)** 467-475.

10. Semeraro S, Mezzanzanica D, Spreafico D, Gagliani M, Re D, Tanaka T, Sidhu SK, & Sano H (2006) Effect of different bur grinding on the bond strength of self-etching adhesives *Operative Dentistry* **31(3)** 317-323.

11. Van Meerbeek B, De Munck J, Mattar D, Van Landuyt K, & Lambrechts P (2003) Microtensile bond strengths of an etch & rinse and self-etch adhesive to enamel and dentin as a function of surface treatment *Operative Dentistry* **28(5)** 647-660.

12. Cardoso MV, Coutinho E, Ermis RB, Poitevin A, Van Landuyt K, De Munck J, Carvalho RCR, & Van Meerbeek B (2008) Influence of dentin cavity surface finishing on micro-tensile bond strength of adhesives *Dental Materials* **24(4)** 492-501.

13. Ogata M, Harada N, Yamaguchi S, Nakajima M, Pereira PNR, & Tagami J (2001) Effects of different burs on dentin bond strengths of self-etching primer bonding systems *Operative Dentistry* **26(4)** 375-382.

14. Inoue H, Inoue S, Uno S, Takahashi A, Koase K, & Sano H (2001) Microtensile bond strength of two single-step adhesive systems to bur-prepared dentin *Journal of Adhesive Dentistry* **3(2)** 129-136.

15. Tani C, & Finger WJ (2002) Effect of smear layer thickness on bond strength mediated by three all-in-one self-etching priming adhesives *Journal of Adhesive Dentistry* **4(4)** 283-289.

16. De Oliveira MT, Di Francescantonio M, Consani S, Giannini M, & Ambrosano GMB (2007) Influence of dentin smear layer created by chemo-mechanical or bur excavation methods on adhesion of self-etching primers and a conventional adhesive *Journal of Adhesion* **83(9)** 821-835.

17. Tay FR, Pashley DH, King NM, Carvalho RM, Tsai J, Lai SCN, & Marquezini L Jr. (2004) Aggressiveness of self-etch adhesives on unground enamel *Operative Dentistry* **29(3)** 309-316.

18. Hashimoto M, Ohno H, Yoshida E, Hori M, Sano H, Kaga M, & Oguchi H (2003) Resin-enamel bonds made with self-etching primers on ground enamel *European Journal of Oral Sciences* **111(5)** 447-453.

19. Pashley DH, & Tay FR (2001) Aggressiveness of contemporary self-etching adhesives. Part II: Etching effects on unground enamel *Dental Materials* **17(5)** 430–444.

20. Tay FR, & Pashley DH (2001) Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers *Dental Materials* **17(4)** 296–308.

21. Adebayo OA, Burrow MF, & Tyas MJ (2008) Bonding of one-step and two-step self-etching primer adhesives to dentin with different tubule orientations *Acta Odontologica Scandinavica* **66(3)** 159-168.

22. de Carvalho RCR, De Freitas PM, Otsuki M, Eduardo CD, & Tagami J (2008) Micro-shear bond strength of Er:YAG-laser-treated dentin *Lasers in Medical Science* **23(2)** 117-124.

23. De Munck J, Van Meerbeek B, Yudhira R, Lambrechts P, & Vanherle G (2002) Micro-tensile bond strength of two adhesives to Erbium:YAG-lased vs. bur-cut enamel and dentin *European Journal of Oral Sciences* **110(4)** 322-329.

24. Dunn WJ, Davis JT, & Bush AC (2005) Shear bond strength and SEM evaluation of composite bonded to Er:YAG laser-prepared dentin and enamel *Dental Materials* **21(7)** 616-624.

25. Lee B-S, Lin P-Y, Chen M-H, Hsieh T-T, Lin C-P, Lai J-Y, & Lan W-H (2007) Tensile bond strength of Er,Cr:YSGG laser-irradiated human dentin and analysis of dentin-resin interface *Dental Materials* **23(5)** 570-578.

26. Lin S, Caputo AA, Eversole LR, & Rizoiu I (1999) Topographical characteristics and shear bond strength of tooth surfaces cut with a laser-powered hydrokinetic system *Journal of Prosthetic Dentistry* **82(4)** 451-455.

27. Tachibana A, Marques MM, Soler JMP, & Matos AB (2008) Erbium, chromium:yttrium scandium gallium garnet laser for caries removal: Influence on bonding of a self-etching adhesive system *Lasers in Medical Science* **23(4)** 435-441.

28. Kim JH, Kwon OW, Kim HI, & Kwon YH (2006) Acid resistance of erbium-doped yttrium aluminum garnet laser-treated and phosphoric acid-etched enamels *Angle Orthodontist* **76(6)** 1052-1056.

29. Hossain M, Nakamura Y, Tamaki Y, Yamada Y, Murakami Y, & Matsumoto K (2003) Atomic analysis and knoop hardness measurement of the cavity floor prepared by Er,Cr:YSGG laser irradiation in vitro *Journal of Oral Rehabilitation* **30(5)** 515-521.

30. Kimura Y, Yu DG, Fujita A, Yamashita A, Murakami Y, & Matsumoto K (2001) Effects of erbium,chromium:YSGG laser irradiation on canine mandibular bone *Journal of Periodontology* **72(9)** 1178-1182.

31. Komet (2007) *Innovations* Komet Lemgo, Vertretung, Germany.

32. Sevgican F, Inoue S, Koase K, Kawamoto C, Ikeda T, & Sano H (2004) Bond strength of simplified-step adhesives to enamel prepared with two different diamond burs *Australian Dental Journal* **49(3)** 141-145.

33. Cardoso MV, De Munck J, Coutinho E, Ermis RB, Van Landuyt K, De Carvalho RCR, & Van Meerbeek B (2008) Influence of ErCr : YSGG laser treatment on microtensile bond strength of adhesives to enamel *Operative Dentistry* **33(4)** 448-455.

34. Adebayo OA, Burrow MF, Tyas MJ, Adams GG, & Collins ML Enamel microhardness and bond strengths of self-etching primer adhesives *European Journal of Oral Sciences* **118(2)** 191-196.

35. Martinez-Insua A, Dominguez LD, Rivera FG, & Santana-Penin UA (2000) Differences in bonding to acid-etched or Er:YAG-laser-treated enamel and dentin surfaces *Journal of Prosthetic Dentistry* **84(3)** 280-288.

36. Ceballos L, Toledano M, Osorio R, Tay FR, & Marshall GW (2002) Bonding to Er-YAG-laser-treated dentin *Journal of Dental Research* **81(2)** 119-122.

37. Ermis RB, De Munck J, Cardoso MV, Coutinho E, Van Landuyt KL, Poitevin A, Lambrechts P, & Van Meerbeek B (2008) Bond strength of self-etch adhesives to dentin prepared with three different diamond burs *Dental Materials* **24(7)** 978-985.

38. Kwong SM, Cheung GSP, Kei LH, Itthagarun A, Smales RJ, Tay FR, & Pashley DH (2002) Micro-tensile bond strengths to sclerotic dentin using a self-etching and a total-etching technique *Dental Materials* **18(5)** 359-369.

39. 3M-ESPE (2007) *Filtek Silorane Low Shrinkage Posterior Restorative, Silorane System Adhesive Self-Etch Primer and Bond* 3M-ESPE, Seefeld, Germany.