Photomedicine and Laser Surgery
Volume 27, Number 1, 2009

Pp. 3–10
DOI: 10.1089/pho.2007.2170

Tensile Bond Strength of Single-Step Self-Etch Adhesives to Er:YAG Laser-Irradiated Dentin

Atsushi Kameyama, D.D.S., Ph.D., Koya Aizawa, D.D.S., Ph.D., Junji Kato, D.D.S., Ph.D., and Yoshito Hirai, D.D.S., Ph.D.

Abstract

Objective: The purpose of this study was to evaluate the influence of Er:YAG laser irradiation on the bond strength to dentine of three single-step adhesives (AQ Bond Plus, G-Bond, and Clearfil Tri-S Bond), and one two-step self-etch adhesive (Clearfil Megabond) as a control. ***Background Data:*** The vast majority of the numerous reports on resin bonding to Er:YAG-lased dentine have concluded that Er:YAG laser irradiation is less effective in terms of bond strength, because of the sub-surface damage it produces. However, its effect in combination with single-step adhesives on bonding to dentine remains to be clarified. ***Methods:*** Eighty bovine incisors were ground with silicon carbide paper to obtain a flat dentine surface, which 40 were irradiated with an Er:YAG laser. Both lased and unlased dentine was bonded to a resin composite with each adhesive. Tensile bond strength was determined after 24 h of storage in water at 37°C. Failure patterns after tensile bond testing was analyzed by scanning electron microscopy. ***Results:*** The two-step self-etch adhesive (Clearfil Megabond) showed the highest bond strength to unlased dentine, but was significantly less effective on lased dentine than the three single-step adhesives. On the other hand, AQ Bond Plus produced an effective bond strength to both lased and unlased dentine, perhaps due to its low viscosity. ***Conclusion:*** The single-step adhesives tested in this study were as effective in combination with Er:YAG-lased dentine as the two-step self-etch adhesive.

Introduction

THE BASIC METHOD FOR ACHIEVING ADHESION between resin and dentine involves three steps: (1) applying an acid to remove the smear layer and slightly demineralize the bonded surface (etch and rinse); (2) modifying the demineralized surface to facilitate infiltration of the resin monomer (priming); and (3) actually applying the adhesive resin to demineralized dentine and then polymerizing to interlock micro-mechanically (bonding).[1,2] Advances in bonding techniques have simplified this procedure, and at present, two-step etch-and-rinse or two-step self-etch adhesives are generally used.

In recent years, single-step self-etch adhesives have been developed and made commercially available. This type of adhesive incorporates the three aforementioned bonding steps into a single step. Single-step adhesives reduce application time and simplify the procedure. Furthermore, with some single-step adhesives, the hybrid layer is almost absent, although the reaction of functional monomers and hydroxyapatite, the so-called "nano-interaction zone," can still be observed.[3]

Resin bonding to Er:YAG laser-irradiated dentine has been extensively reported on.[4–8] Such Er:YAG-lased dentine typically has a porous, imbricate patterned surface, and no smear layer.[9] Most researchers have studied two- or three-step total-etch adhesives or two-step self-etch adhesives, with almost all concluding that Er:YAG laser irradiation was less effective in terms of bond strength[5–8] since it causes sub-surface damage.[10] In contrast, the bond strength between Er:YAG-laser-irradiated dentine and a single-step adhesive has not yet been adequately evaluated.

The purpose of this study was to evaluate the influence of Er:YAG laser irradiation on the bond strength to dentine of three single-step adhesives and one two-step self-etch adhesive as a control.

Materials and Methods

The Er:YAG laser equipment used in this study was the Erwin AdvErL (J. Morita Mfg. Corp., Kyoto, Japan) at a wavelength of 2.94 μm. The output energy and pulse repetition rate of this laser device can be varied from 30 to 250 mJ per pulse and 1 to 25 pulses per second (pps). However,

Department of Operative Dentistry, Tokyo Dental College, Chiba, Japan.

TABLE 1. ADHESIVES USED IN THIS STUDY

Code	Adhesive (manufacturer)	Main components	pH[a]	Batch no.	Resin composite (manufacturer)
Single-step self-etch adhesives					
AQP	AQ Bond Plus (Sun Medical)	Liquid: water, acetone, 4-META, UDMA, HEMA, MMA, initiator	2.5	KE1	Metafil C (Sun Medical)
		Eponge: p-toluensulfinic sodium salt		LT1	
GB	G-Bond (GC)	4-MET, UDMA, acetone, water, silanated colloidal silica, initiator	2.0	0510201	Solare (GC)
TS	Clearfil Tri-S Bond (Kuraray Medical)	MDP, HEMA, Bis-GMA, water, ethanol, photoinitiator, silinated colloidal silica	2.7	011159	Clearfil AP-X (Kuraray Medical)
Two-step self-etch adhesive					
MB	Clearfil Megabond[b] (Kuraray Medical)	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate photoinitiator, aromatic tertiary amine, water	1.9	01014A	Clearfil AP-X (Kuraray Medical)
		Bond: 10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, photoinitiator, aromatic tertiary amine, silanated colloidal silica	1.6		

[a]According to Sasakawa et al.[35] and Koshiro et al.[3]
[b]Also known as Clearfil SE Bond in Europe and USA
4-META, 4-metacryloxyethyl trimellitic acid; UDMA, urethane dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; 4-MET, 4-metacryloxyethyl trimellitic acid; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-glycidyl methacrylate

TABLE 2. APPLICATION PROTOCOLS OF THE ADHESIVE SYSTEMS TESTED

Adhesive	Application protocol
AQP	Dispense one drop of liquid into well containing one piece of sponge (Eponge); apply mixed Eponge to dentine for 20 sec; gently ar dry for 5–10 sec and light cure for 10 sec
GB	Apply sufficient amount of adhesive for 10 sec; briskly air dry and light cure for 10 sec
TS	Apply sufficient amount of adhesive for 20 sec; briskly air dry and light cure for 10 sec
MB	Apply primer for 20 sec and gently air dry; immediately after, apply bond; mildly air dry and light cure for 10 sec

TABLE 3. VISCOSITY (CENTIPOISE [CP]) AND TENSILE BOND STRENGTH (MEAN ± SD, MPA) FOR EACH GROUP

Adhesive	AQP	GB	TS	MB
Viscosity	3.6	48.6	150.0	441.3
Tensile bond strength				
Lased	10.8 ± 1.4^{a}	8.7 ± 1.8^{b}	8.4 ± 1.8^{bc}	7.0 ± 2.1^{c}
p Value	0.1288 (NS)	0.042 (S)	0.0091 (S)	<0.0001 (S)
Unlased	12.1 ± 1.6^{ab}	11.2 ± 1.8^{b}	10.6 ± 1.6^{b}	13.4 ± 2.7^{a}

Mean values designated with same letter were not significantly different (Fisher's PLSD; $p > 0.05$).
Each p value indicates significant difference between lased and unlased groups (S, significant, NS, not significant).

the total energy is limited to a maximum of approximately 1.2 W at the end of the probe. In this study, the laser was adapted with a contact tip with a 600-μm diameter, and the end of the probe was then set at 100 mJ/pulse and 10 pps. The pulse duration of the laser was set at 400 μs. Energy levels were measured on demand with a power meter (LaserMate-P; Coherent Inc., Santa Clara, CA, USA).

The four combinations of adhesive system/resin composites investigated in this study and described in Table 1 were as follows: AQ Bond Plus/Metafil C (Sun Medical, Moriyama, Shiga, Japan), G-Bond/Solare (GC, Tokyo, Japan), Clearfil Tri-S Bond/Clearfil AP-X (Kuraray Medical, Osaka, Japan), and Clearfil Megabond/Clearfil AP-X (Kuraray Medical).

Eighty bovine incisors, frozen to maintain freshness and defrosted immediately before specimen preparation, were used in this study. Labial surfaces of the teeth were ground under a stream of water with silicon carbide paper up to 180-grit to produce a flat dentine surface. Of these, 40 dentine surfaces were then uniformly irradiated with the Er:YAG laser under a water spray (4 mL/min) during operation by a freehand technique.[11] The tip of this laser was placed in light contact with the dentine to allow free movement. The 40 Er:YAG-lased and 40 unlased dentine surfaces were then

Table 4. Failure Patterns in Tensile Bond-Tested Samples as Analyzed through Stereo-Microscopy

		Type 1 *100% R*	*Type 2* *mixed R > I, D*	*Type 3* *Mixed I, D > R*	*Type 4* *100% I*	*Total*
AQP	Lased	0	5	5	0	10
	Unlased	0	8	2	0	10
GB	Lased	0	2	8	0	10
	Unlased	0	4	6	0	10
TS	Lased	0	4	6	0	10
	Unlased	0	0	8	2	10
MB	Lased	0	3	7	0	10
	Unlased	0	0	9	1	10

R, failure within resin composite or adhesive resin; I: interfacial failure between dentine and adhesive; D: failure in dentine.

randomly divided into four experimental subgroups (n = 10 each).

Double-sided adhesive tape with a 4.8-mm diameter hole was attached to the flattened dentine surface. The primer (only in MB) and the adhesives were applied to the dentine surface area through the hole in the adhesive tape, followed by light curing with a quartz-tungsten-halogen curing unit (Candelux; J. Morita Mfg. Co.) according to each manufacturer's instructions, as shown in Table 2. After the bonding procedure, a piece of 0.7-mm-thick cardboard with a 4.8-mm diameter hole was aligned with and affixed to the adhesive tape, and the mold was filled with resin composite and light-cured for 20 sec. After a PMMA rod was attached to the cured composite with 4-META/MMA-TBB resin (Superbond C&B; Sun Medical), the bonded specimen was immersed in 37°C water for 24 h, and the tensile bond strength (TBS) was then

A B C

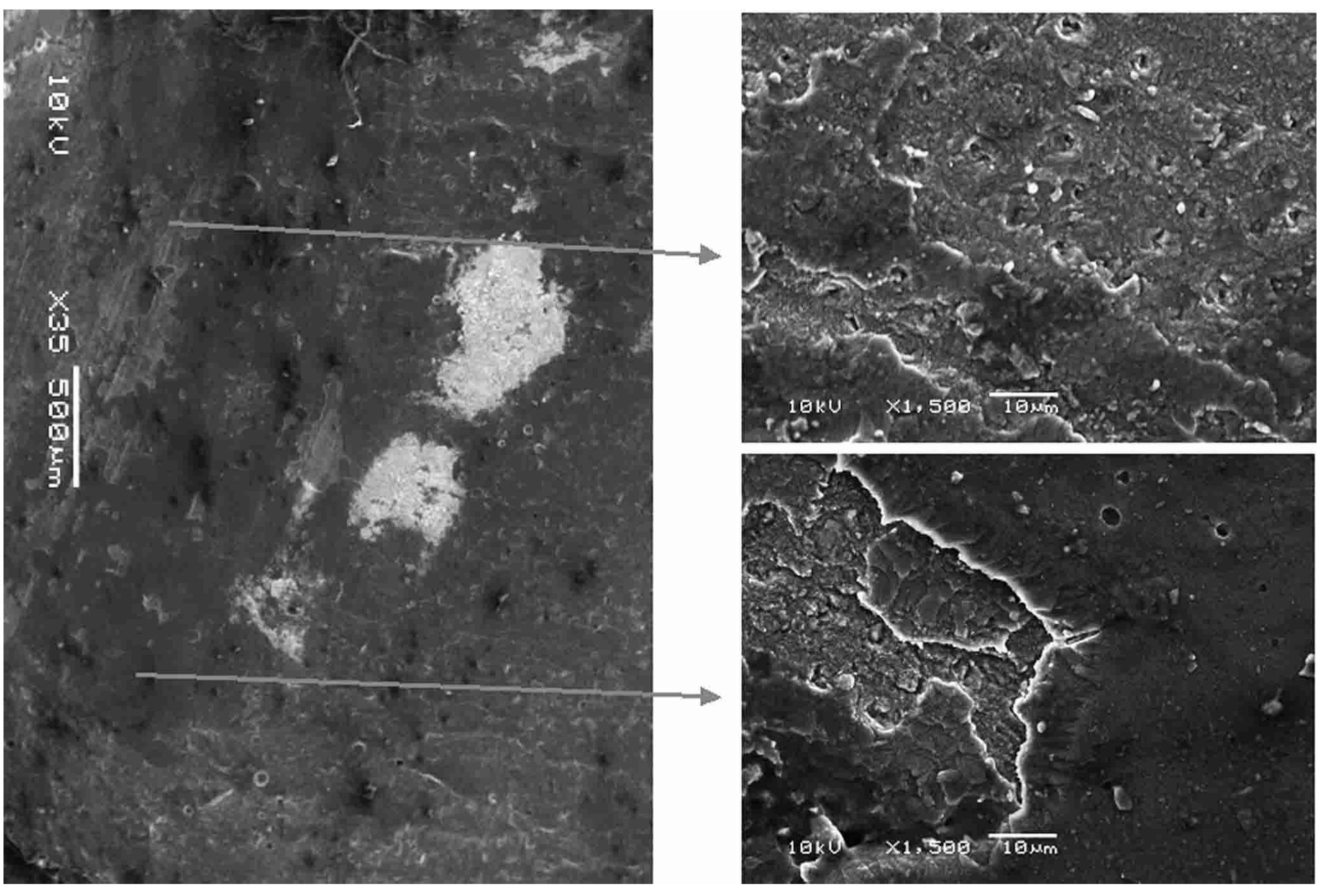

FIG 1. SEM micrographs of the dentine side of a fractured surface from the unlased AQP group. (**A**) Low magnification (original magnification 35×) revealed a variety of cohesive failures in adhesive resin and adhesive failure at adhesive-dentine interface. (**B**) High magnification (1500×) revealed the main area of failure at the adhesive interface, with dentinal tubules clearly visible, together with fractured resin tags inside. (**C**) High magnification (1500×) revealed the main area of cohesive failure in adhesive/resin, with fractured resin tags present within dentinal tubules.

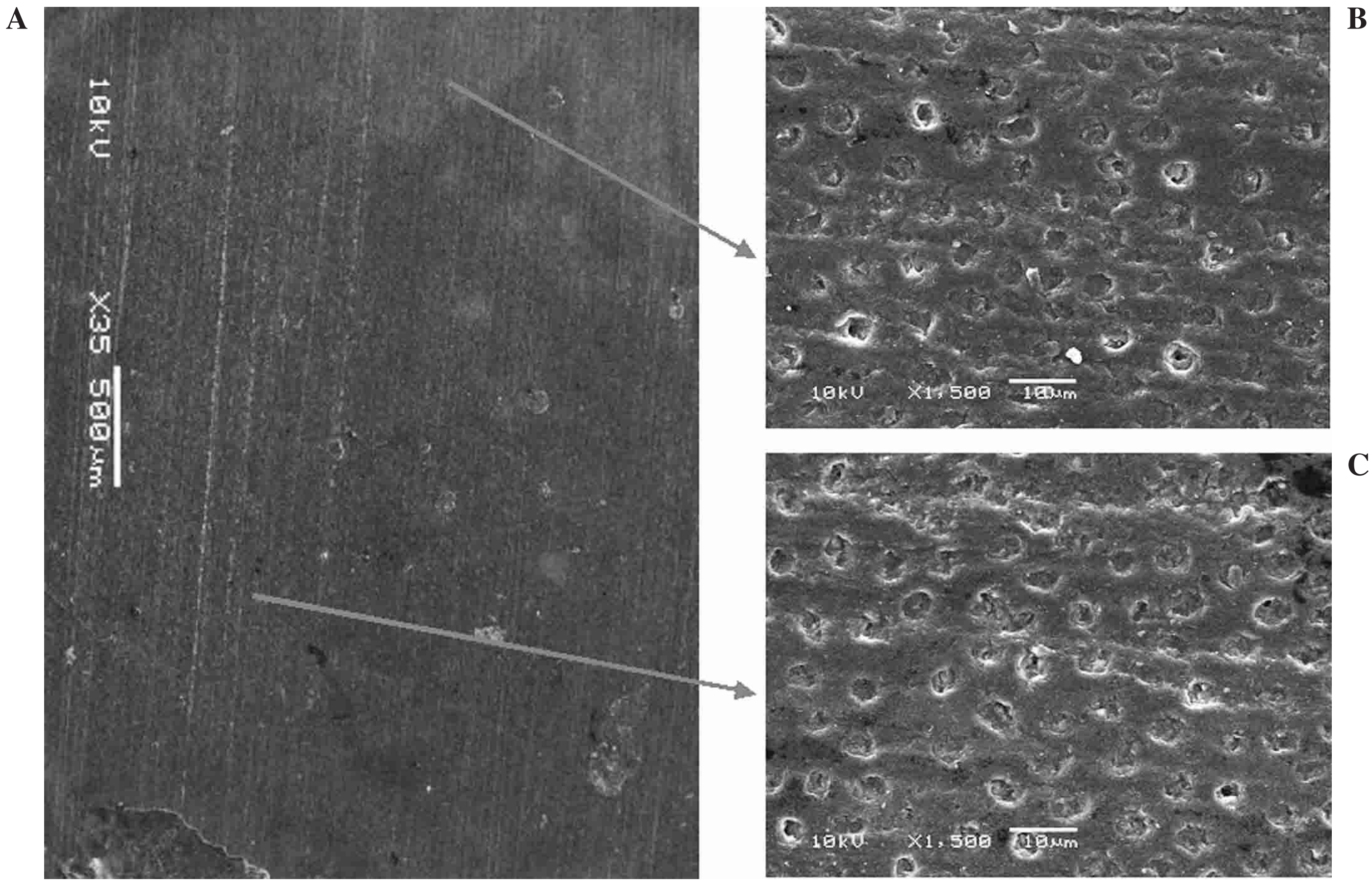

FIG 2. SEM micrographs of the dentine side of a fractured surface from the unlased-MB group. (**A**) Low magnification (35×) showing adhesive interface, with scratches caused by the silicon carbide paper more clearly visible than in Fig. 3. (**B** and **C**) High magnification (1500×) revealed fractured resin tags plugging the dentinal tubules.

tested using a universal testing machine (Shimadzu, Kyoto, Japan) at a cross-head speed of 2.0 mm/min.

After TBS measurement, failure modes were classified using a stereomicroscope at 50× magnification. Failure mode was categorized as one of four types: type 1: 100% cohesive failure in resin composite or adhesive resin; type 2: mixed failure, mainly within resin composite/adhesive resin, but partially within adhesive interface and/or dentine; type 3: mixed failure, mainly within dentine and/or adhesive interface, but partially within resin composite; and type 4: failure in adhesive interface (includes partial cohesive failure in dentine). Additionally, samples exhibiting the representative failure mode and a TBS close to the average value were selected from each group and examined by scanning electron microscopy (SEM) (JSM-5610LV; JEOL, Tokyo, Japan). The specimens were dehydrated in ascending grades of ethanol, dried in a desiccator for 1 d, and Pt-sputter coated with the Super Fine Coater (ESC-101; Elionix, Hachioji, Tokyo, Japan) for 200 sec before SEM examination.

The tensile bond strength of each specimen was recorded and subjected to one-way and two-way ANOVAs. Differences were considered statistically significant at $p < 0.05$. All analyses were carried out using a commercially available statistical package (StatView 5.0; SAS Institute, Cary, NC, USA).

The viscosity of each adhesive was also measured. One milliliter of adhesive was instilled into an E-type viscosity analyzer (Visconic EHD; Tokimec, Tokyo, Japan) at 15°C, and measured at 100 rpm. Measurements were performed once for each adhesive.

Results

The viscosity of each adhesive and TBS in each group are summarized in Table 3. The two-way ANOVA revealed a significant interaction between pairs of means for "laser irradiation" ($p < 0.0001$) and "adhesive system" ($p = 0.0097$). There was a significant interaction between the independent variables of "laser irradiation" and "adhesive system" ($p = 0.0003$). Therefore, multiple comparisons among all tested groups were performed using Fisher's protected least significant difference (PLSD) test at the 5% significance level.

When bonded to unlased dentine, of the four adhesives, MB produced the highest bond strength, with a significant difference observed between TS ($p = 0.0015$) and GB ($p = 0.0091$). However, no significant difference was observed between MB and AQP ($p = 0.0801$).

When bonded to Er:YAG-lased dentine, of the four adhesives, AQP produced the highest bond strength, which was also significantly higher than that of the other three adhesives (versus GB: $p = 0.0125$; versus TS: $p = 0.0047$; versus MB: $p < 0.0001$).

When comparing lased and unlased dentine, only AQP

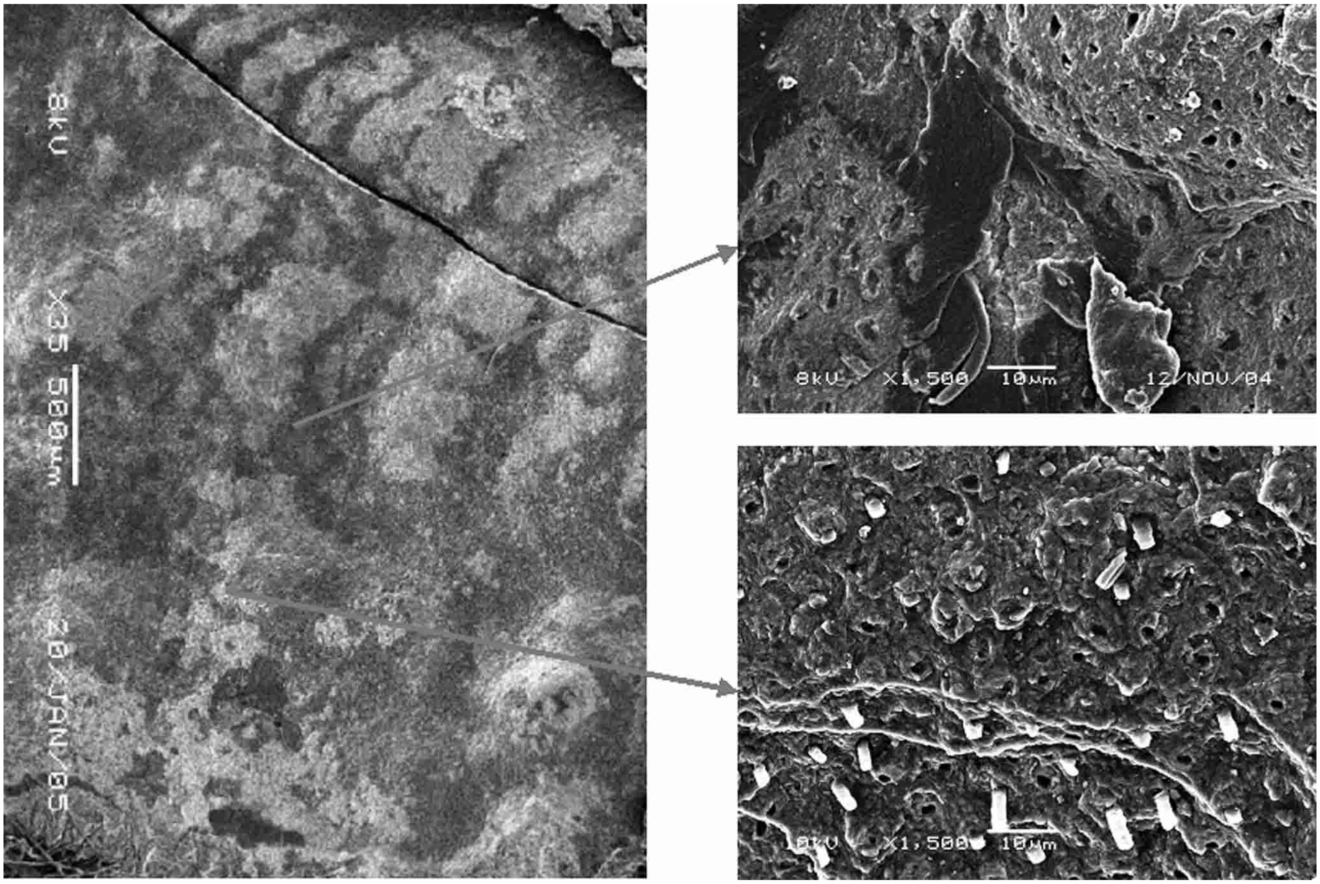

FIG. 3. SEM micrographs of dentine-side of a fractured surface from the Er:YAG lased-AQP group. (**A**) Low magnification (35×). (**B**) High magnification (1500×); here the failure patterns showed cohesive failure in laser-affected dentine, adhesive resin, and partially within the hybrid layer. Adhesive resin tags were seen in the dentinal tubules in the hybrid-layer-failed area. (**C**) High magnification view of other areas (1500×), showing cohesive failure in laser-affected dentine, with fractured resin tags remaining in the dentinal tubules.

showed no significant difference ($p = 0.1288$). The other three adhesives showed significantly lower bond strengths to Er:YAG-lased dentine than unlased dentine ($p < 0.05$).

The respective modes of failure are summarized in Table 4, and SEM views of fractured surfaces are shown in Figs. 1–4. In unlased dentine, most AQP specimens showed mainly cohesive failure in the adhesive resin (Fig. 1), but the other three adhesives mostly showed mixed failure, mainly at the adhesive interface, with partial cohesion in the adhesive resin (Fig. 2). In the lased dentine, all specimens showed mixed failure in each adhesive; and most specimens in each group showed failure mainly at the adhesive interface or laser-affected dentine, although the number of specimens showing type 2 and type 3 failure was even for AQP (Figs. 3 and 4).

Discussion

Several factors have been reported to affect the quality of adhesion to lased dentine, including output energy,[12,13] pulse duration,[14,15] focal distance between the tip and the dentine surface,[16] the adhesive system used,[6,17] acid etching,[7,18] and additional priming.[12,19] In order to irradiate uniformly, some studies employed laser irradiation with the dentine specimen fixed to a moving stage.[11,12,20] However, this study employed freehand irradiation, since our previous data indicated no significant difference between uniform irradiation using a moving stage and freehand irradiation.[11]

In this study, four commercially available adhesives were used. There have already been several reports on the bonding properties of these adhesives to unlased dentine. In this study, MB was selected as the control. This adhesive is well known as one of the most reliable two-step self-etch adhesive systems due to its simplicity of use, good and stable clinical performance, reduced technical sensitivity, mechanical strength, and consistent composition.[2,21–23] Furthermore, many *in vitro* studies have verified its high bond strength to both enamel and dentine.[1,24,25] In the present study, MB also showed the highest bond strength to unlased dentine among the four adhesives tested. This result supports those of a study by Ishikawa et al.,[26] which evaluated the same four adhesives for both micro-tensile and micro-shear bond strength. In a recent study, 10-MDP, the functional monomer included in MB, was found to interact chemically with calcium in hydroxyapatite more effectively than other monomers, which might also contribute to its high bond strength.[27] Additionally, its stronger mechanical properties and polymerization efficacy may also contribute to its higher bond strength.[28,29]

A B C

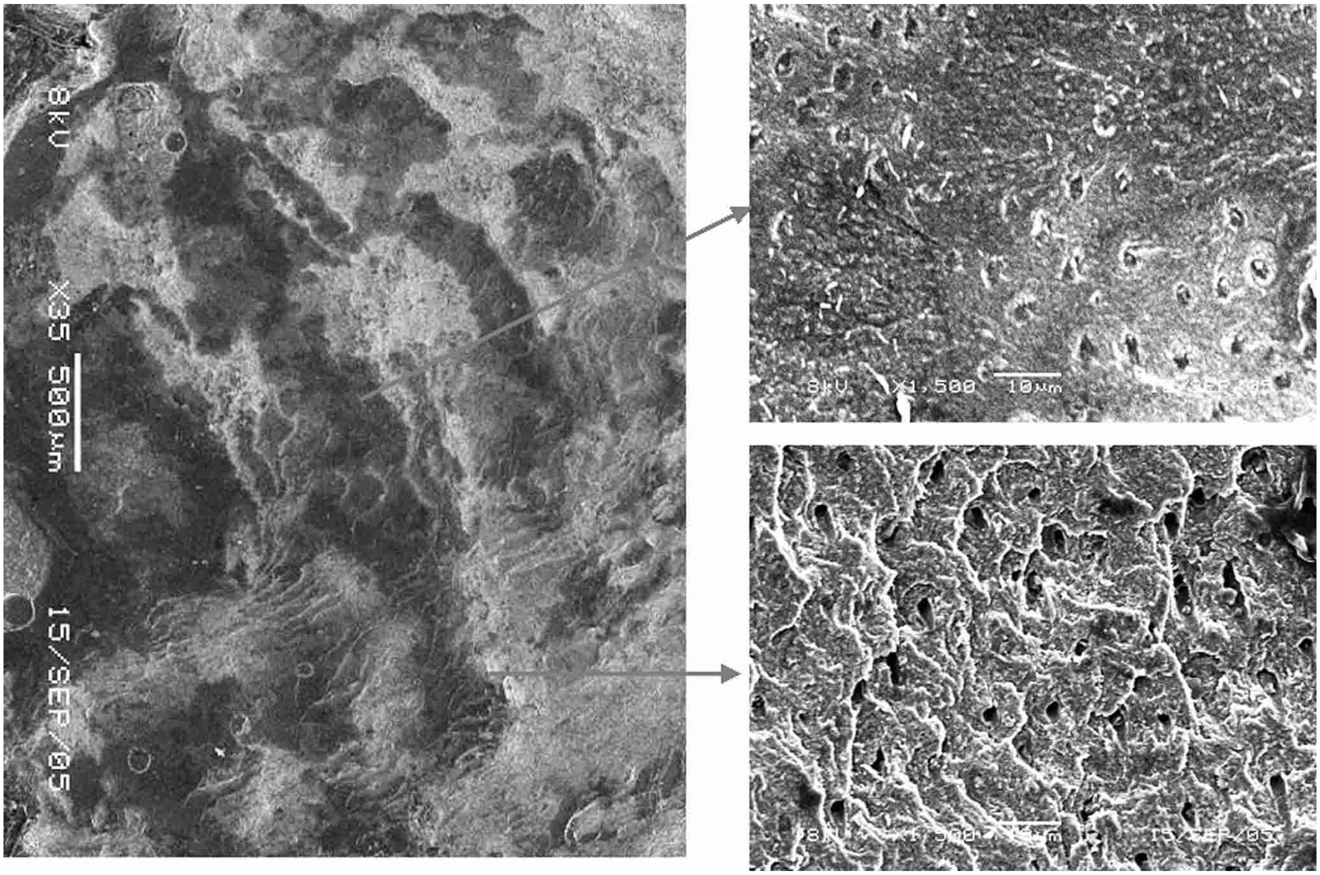

FIG. 4. SEM micrographs of the dentine side of a fractured surface from the Er:YAG lased-MB group. (**A**) Low magnification (35×). (**B** and **C**) High magnification (1500×), showing cohesive failures within the hybrid layer (**B**) and dentine (**C**), with fractured resin tags not seen in areas of dentinal failure.

AQP, GB, and TS are one-bottle, single-step self-etch adhesives, or so-called “all-in-one” adhesives. To date, single-step adhesives have generally achieved a lower bond strength than multi-step adhesives[2] due to a number of unfavorable features: they inhibit water movement across the adhesive layer due to their high hydrophilicity;[30] they show reticular patterns of nano-leakage, the so-called “water trees”;[31] they form voids within the adhesive layer;[32] and HEMA-free single-step adhesives show phase separation.[33,34] In the present study, however, AQP showed no significant difference in comparison with the MB control. Sasakawa et al.[35] also compared the micro-shear bond strengths of five single-step adhesives with MB, and found that only AQP showed a high bond strength, equivalent to that of MB. The acidic monomers comprising AQP slightly demineralize superficial dentine, forming a very thin ($\sim$1 μm) hybrid layer,[3,35] and AQP’s morphological characteristics are similar to those of MB.

On the other hand, the tensile bond strengths of GB and TS were significantly lower than those of both AQP and MB. All three single-step adhesives investigated in this study are categorized as “mild” self-etch adhesives, because they have a pH of more than 2. However, Koshiro et al.[3] reported that neither GB nor TS showed a typical hybrid layer in a TEM study in which the dentine was ground with 600-grit silicon carbide paper. The decalcification abilities of GB and TS may therefore be quite small. In contrast to the method of Koshiro’s group, we ground the adhered dentine with #180-grit silicon carbide. Therefore the smear layer created may have been thicker than that achieved with 600-grit, which may have interfered with monomer penetration into the superficial dentine.

The tensile bond strengths of adhesives to Er:YAG-lased dentine were significantly lower than those to unlased dentine, except for AQP. Of note is the result that the tensile bond strength of MB to Er:YAG-lased dentine was about half of that to unlased dentine. De Munck et al.[6] also compared micro-tensile bond strength between Er:YAG-lased and diamond bur-cut dentine, with MB and the three-step total-etch adhesive OptiBond FL (Kerr, Orange, CA, USA). Even though both adhesives were found to be quite reliable, the tensile bond strength of Er:YAG-lased dentine was very low compared to that of bur-cut dentine. The Er:YAG laser creates subsurface damage in the form of deep cracks (about 20–60 μm),[10] and adhesive monomers are not able to penetrate sufficiently into the damaged surface.

AQP showed the highest bond strength among the four adhesives in the lased group, with no significant difference seen between the lased and unlased groups. AQP has a more complicated structure than the other adhesives and its viscosity is quite low (Table 3), which may enable it to easily penetrate laser-affected dentine, which has micro-cracks and laser-modified organic components. In this study, adhesives with high viscosity tended to show low bond strength. It is therefore suggested that the viscosity of the adhesive is one of the factors that influences the strength of the bond to Er:YAG-lased dentine.

Each debonded surface showed a variety of failures in the adhesive interface and cohesive failures in the resin composite/adhesive resin in the unlased group (Figs. 3 and 4). AQP showed mostly mixed failure, mainly involving the adhesive resin, probably due to the weaker mechanical properties of the adhesive resin itself. In contrast, numerous dentinal tubules were seen in the MB and TS samples. These may have been due to the stronger mechanical properties of the adhesive resins. In contrast, numerous dentinal tubules were seen in the MB and TS samples. These may have been due to the stronger mechanical properties of the adhesive resins. Therefore, they should be interpreted as adhesive failures at the resin–dentin interface. Numerous dentinal tubules were also observed in the Er:YAG-lased groups. However, they should not be interpreted as adhesive failures, but as the cohesive failures in the laser-damaged dentin which were not impregnated by adhesive resin.[5,14] To achieve sufficient adherence to Er:YAG-lased dentine, it is necessary for the resin monomer to penetrate the laser-affected dentine subsurface to a depth of more than 15 μm.[36] In AQP, a larger area of cohesive failure in the adhesive resin was observed than with the other three adhesives. This suggests that the viscosity of the adhesive resin is an important factor in bonding to Er:YAG-lased dentine.

GB in the lased group also fractured cohesively within most parts of the dentine, suggesting insufficient penetration of the adhesive into the laser-affected area (data not shown). In an earlier study, we demonstrated the effect of HEMA on bonding to lased dentine.[18] However, GB does not contain HEMA, and this is one of the reasons for the low bond strength it exhibits. In addition, numerous voids were seen in the fractured adhesive area (data not shown), a phenomenon that led to monomer-solvent phase separation.[32–34] Generally, thorough air-drying of the adhesive prevents this phenomenon, which suggests difficulty in removing interfacial water droplets within the lased dentine.

This study revealed a tendency for single-step adhesives to show a somewhat higher bond strength than a two-step self-etch adhesive. Generally, single-step adhesives contains higher concentrations of solvents than multi-step adhesives. While this may cause incomplete resin polymerization or the formation of voids[32] within the adhesive layer in unlased dentine, it appeared to result in lower viscosity. This may have allowed the adhesive to permeate the laser-affected dentine area, thus resulting in high bond strength to the Er:YAG-lased dentine. In an earlier study, we found no significant difference in tensile bond strength between Er:YAG-lased enamel and unlased enamel when using the single-step adhesives tested in this study.[37] Taken together with the results of this study, this indicates that these adhesives may perform as well as contemporary two-step self-etch adhesives in a clinical setting.

Conclusion

In this study, Er:YAG laser irradiation adversely affected tensile bond strength in the GB, TS, and MB adhesives, but not in AQP adhesive. Furthermore, AQP showed the highest bond strength.

Acknowledgments

The authors would like to thank Associate Professor Jeremy Williams, Tokyo Dental College, for his assistance with the English review of the manuscript.

Disclosure Statement

No competing financial interests exist.

References

1. De Munck, J., Van Landuyt, K., Peumans, M., et al. (2005). A critical review of the durability of adhesion to tooth tissue: methods and results. J. Dent. Res. 84, 118–132.
2. Van Meerbeek, B., Van Landuyt, K., De Munck, J., et al. (2005). Technique-sensitivity of contemporary adhesives. Dent. Mater. J. 24, 1–13.
3. Koshiro, K., Sidhu, S.K., Inoue, S., Ikeda, T., and Sano, H. (2006). New concept of resin-dentin interfacial adhesion: the nanointeraction zone. J. Biomed. Mater. Res. Part B Appl. Biomater. 77B, 401–408.
4. Visuri, S.R., Gilbert, J.L., Wright, D.D., Wigdor, H.A., and Walsh, J.T., Jr. (1996). Shear strength of composite bonded to Er:YAG laser-prepared dentin. J. Dent. Res. 75, 599–605.
5. Kameyama, A., Kawada, E., Takizawa, M., Oda, Y., and Hirai, Y. (2001). Influence of different acid conditioners on the tensile bond strength of 4-META/MMA-TBB resin to Er:YAG laser-irradiated bovine dentin. J. Adhes. Dent. 2, 297–304.
6. De Munck, J., Van Meerbeek, B., Yudhira, R., Lambrechts, P., and Vanherle, G. (2002). Micro-tensile bond strength of two adhesives to erbium:YAG-lased vs. bur-cut enamel and dentin. Eur. J. Oral Sci. 110, 322–329.
7. Ceballos, L., Toledano, M., Osorio, R., Tay, F.R., and Marshall, G.W. (2002). Adhesion of Er-YAG-laser-treated dentin. J. Dent. Res. 81, 119–122.
8. Çelik, E.U., Ergücü, Z., Türkün, L.S., and Türkün, M. (2006). Shear bond strength of different adhesives to Er:YAG laser-prepared dentin. J. Adhes. Dent. 8, 319–325.
9. Kameyama, A., Kawada, E., Takizawa, M., et al. (2000). Influence of acid treatment on Er:YAG laser-irradiated bovine dentin: morphological changes of lased surface. Adhes. Dent. 18, 126–132 (in Japanese).
10. Ishizaka, Y., Eguro, T., Maeda, T., and Tanaka, H. (2002). Effects of Er:YAG laser irradiation on human dentin: polarizing microscopic, light microscopic and microradiographic observations, and FT-IR analysis. Lasers Surg. Med. 31, 171–176.
11. Aizawa, K., Kameyama, A., Kato, J., Oda, Y., and Hirai, Y. (2008). Influence of free hand vs. uniform irradiation on tensile bond strength in Er:YAG-lased dentin. J. Adhes. Dent. 10, 295–299.
12. Mine, A. (2003). Effect of Er:YAG laser irradiation on bonding, structural and mechanical properties of dental hard tissues. J. Okayama Dent. Assoc. 22, 123–137 (in Japanese).
13. Monghini, E.M., Wanderley, R.L., Pecora, J.D., Palma Dibb, R.G., Corona, S.A.M., and Borsatto, M.C. (2004). Bond strength to dentin of primary teeth irradiated with varying Er:YAG laser energies and SEM examination of the surface morphology. Lasers Surg. Med. 34, 254–259.
14. Aizawa, K., Kameyama, A., Kato, J., et al. (2006). Resin bonding to dentin irradiated by high repetition rate Er:YAG laser. Photomed. Laser Surg. 24, 397–401.

15. Gonçalves, M., Corona, S.A.M., Borsatto, M.C., Pecora, J.D., and Palma Dibb, R.G. (2005). Influence of pulse frequency of Er:YAG laser on the tensile bond strength of a composite to dentin. Am. J. Dent. 18, 165–167.
16. de Souza-Gabriel, A.E., Chinelatti, M.A., Borsatto, M.C., Pecora, J.D., Palma-Dibb, R.G., and Corona, S.A.M. (2006). Effect of Er:YAG laser irradiation distance on superficial dentin morphology. Am. J. Dent. 19, 217–221.
17. Ramos, R.P., Chimello, D.T., Chinelatti, M.A., Nonaka, T., Pecora, J.D., and Palma-Dibb, R.G. (2002). Effect of Er:YAG laser on bond strength to dentin of a self-etching primer and two single-bottle adhesive systems. Lasers Surg. Med. 31, 164–170.
18. Kataumi, M., Nakajima, M., Yamada, T., and Tagami, J. (1998). Tensile bond strength and SEM evaluation of Er:YAG laser irradiated dentin using dentin adhesive. Dent. Mater. J. 17, 125–138.
19. Kameyama, A., Kawada, E., Amagai, T., Takizawa, M., Oda, Y., and Hirai, Y. (2002). Effect of HEMA on bonding of Er:YAG laser-irradiated bovine dentine and 4-META/MMA-TBB resin. J. Oral Rehabil. 29, 749–755.
20. Omae, M., Inoue, M., Itota, T., et al. (2007). Effect of a desensitizing agent containing glutaraldehyde and HEMA on bond strength to Er:YAG laser-irradiated dentine. J. Dent. 35, 398–402.
21. Peumans, M., Kanumilli, P., De Munck, J., Van Landuyt, K., Lambrechts, P., and Van Meerbeek, B. (2005). Clinical effectiveness of contemporary adhesives: a systematic review of current clinical trials. Dent. Mater. 21, 864–881.
22. Takahashi, A., Sato, Y., Uno, S., Pereira, P.N.R., and Sano, H. (2002). Effect of mechanical properties of adhesive resins on bond strength to dentin. Dent. Mater. 18, 263–268.
23. Schulze, K.A., Oliveira, S.A., Wilson, R.S., Gansky, S.A., Marshall, G.W., and Marshall, S.J. (2005). Effect of hydration variability on hybrid layer properties of a self-etching versus an acid-etching system. Biomaterials 26, 1011–1018.
24. Inoue, S., Koshiro, K., Yoshida, Y., et al. (2005). Hydrolytic stability of self-etch adhesives bonded to dentin. J. Dent. Res. 84, 1160–1164.
25. Toledano, M., Osorio, R., de Leonardi, G., Rosales-Leal, J.I., Ceballos, L., and Cabrerizo-Vilchez, M.A. (2001). Influence of self-etching primer on the resin adhesion to enamel and dentin. Am. J. Dent. 14, 205–210.
26. Ishikawa, A., Shimada, Y., Foxton, R.M., and Tagami, J. (2007). Micro-tensile and micro-shear bond strengths of current self-etch adhesives to enamel and dentin. Am. J. Dent. 20, 161–166.
27. Yoshida, Y., Nagakane, K., Fukuda, R., et al. (2004). Comparative study on adhesive performance of functional monomers. J. Dent. Res. 83, 454–458.
28. Takahashi, A., Sato, Y., Uno, S., Pereira, P.N.R., and Sano, H. (2002). Effects of mechanical properties of adhesive resins on bond strength to dentin. Dent. Mater. 18, 263–268.
29. Kanehira, M., Finger, W.J., Hoffmann, M., Endo, T., and Komatsu, M. (2006). Relationship between degree of polymerization and enamel bond strength with self-etching adhesives. J. Adhes. Dent. 8, 211–216.
30. Tay, F.R., Pashley, D.H., Suh, B.I., Carvalho, R.M., and Itthagarun, A. (2002). Single-step adhesives are permeable membranes. J. Dent. Res. 30, 371–382.
31. Tay, F.R., and Pashley, D.H. (2003). Water treeing—a potential mechanism for degradation of dentin adhesives. Am. J. Dent. 16, 6–12.
32. Yoshida, E., and Uno, S. (2004). Void formation along the bonding interface between a smeared dentin surface and all-in-one adhesives. Dent. Mater. J. 23, 643–649.
33. Van Landuyt, K.L., De Munck, J., Snauwaert, J., et al. (2005). Monomer-solvent phase separation in one-step self-etch adhesives. J. Dent. Res. 84, 183–188.
34. Hiraishi, N., Breschi, L., Prati, C., Ferrari, M., Tagami, J., and King, N.M. (2007). Technique sensitivity associated with air-drying of HEMA-free, single-bottle, one-step self-etch adhesives. Dent. Mater. 23, 498–505.
35. Sasakawa, W., Nakaoki, Y., Nagano, F., et al. (2005). Micro-shear bond strength of five single-step adhesives to dentin. Dent. Mater. J. 24, 617–627.
36. Aizawa, K., Kameyama, A., Kato, J., and Hirai, Y. (2008). Nano-hardness of adhesive interface between Er:YAG laser-irradiated dentin and 4-META/MMA-TBB resin. Photomed. Laser Surg. 26, 107–111.
37. Kameyama, A., Kato, J., Aizawa, K., Suemori, T., and Hirai, Y. (2008). Tensile bond strength of one-step self-etch adhesives to Er:YAG laser-irradiated and non-irradiated enamel. Dent. Mater. J. 27, 386–391.

Address reprint requests to:
Dr. Atsushi Kameyama, D.D.S., Ph.D.
Department of Operative Dentistry
Tokyo Dental College
1-2-2 Masago, Mihama-ku
Chiba 261-8502, Japan

E-mail: kameyama@tdc.ac.jp