Influence of Free-hand vs Uniform Irradiation on Tensile Bond Strength in Er:YAG-Lased Dentin

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Purpose: To compare effects of free-hand and mechanically applied uniform irradiation on tensile bond strength of 4-META/MMA-TBB resin to Er:YAG laser-irradiated dentin using an X-Y moving stage.

Materials and Methods: Three different laser conditions were evaluated: 1.0 W, 100 mJ/pulse, 10 pps; 1.0 W, 50 mJ/pulse, 20 pps; 1.0 W, 33 mJ/pulse, 30 pps. Samples of bovine dentin were set on a moving stage and mechanically irradiated with an Er:YAG laser at a scanning speed of 1.0 mm/s or subjected to free-hand irradiation. The lased dentin surfaces were acid conditioned with 10% citric acid/3% ferric chloride for 15 s, rinsed, and dried. The surfaces were bonded to PMMA rods with 4-META/MMA-TBB resin, and mini-dumbbell-shaped bonded specimens were prepared. Tensile bond strength was measured and compared with the results for free-hand irradiated samples obtained under the same conditions in an earlier study. SEM observations of fractured surfaces after tensile bond testing were also performed.

Results: A significant difference was found in tensile bond strength among the 3 laser settings in both free-hand and mechanically uniform irradiations. A comparison of free-hand laser irradiation and mechanically performed uniform laser irradiation revealed no significant difference under either laser condition ($p > 0.05$). SEM observation of the fractured surfaces showed no difference between the two irradiation methods under all laser conditions.

Conclusion: No significant difference was found in tensile bond strength between free-hand and mechanically irradiated dentin. Significant differences were found, however, with changes in laser setting in both groups.

Keywords: Er:YAG laser, dentin bonding, 4-META/MMA-TBB resin, laser energy, pulse frequency, output energy.

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Resin bonding to Er:YAG laser-irradiated dentin has already been examined.[5] Most studies have found a decrease in bond strength to lased dentin compared with unlased dentin. This is probably due to a combination of structural and morphological changes in the lased surface. The Er:YAG-lased dentin surface has many unique characteristics: no smear layer, open dentinal tubules, and a flaky and rough surface. While no change in crystal composition has been observed, organic components have been demonstrated to be denatured or disappear.[2,9,16,22,23]

Several factors are believed to affect the quality of adhesion to lased dentin, including output energy,[26,35] the adhesive system used,[4,6,29,34] acid etching,[17,21] and additional priming.[19,35] Few of these studies employed uniform laser irradiation with the dentin specimen fixed to a moving stage.[36] However, these studies have focused on the effect of uniform irradiation in detail, even though some have investigated the influence of focal distance from the tip and the tooth surface.[8,12,29] Our previous studies on resin bonding to Er:YAG-lased dentin[1,17-19] also employed free-hand irradiation.

This study investigated the effect of mechanically controlled uniform irradiation at a constant output energy/rep-

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Table 1 Tensile bond strength (TBS; mean ± SD, MPa) and CV values (%)

Group	Output energy	Pulse frequency	Total energy	Uniform	Statistical significance*	Free-hand**
100-10	100 mJ/pulse	10 pps	1.0 W	17.9 ± 3.3[a] (18.4%)	NS	16.6 ± 4.4[a] (26.5%)
50-20	50 mJ/pulse	20 pps	1.0 W	11.4 ± 4.4[b] (38.6%)	NS	10.7 ± 5.1[b] (47.7%)
33-30	33 mJ/pulse	30 pps	0.99 W	9.9 ± 3.7[b] (37.9%)	NS	9.9 ± 1.6[b] (16.2%)

* Difference between mechanically uniform and free-hand at same laser setting ($p < 0.05$). Same superscript letters indicate no significant difference ($p < 0.05$)
** According to Aizawa et al[1]

etition rate on tensile bond strength to Er:YAG laser-irradiated dentin, and compared the results with those obtained by free-hand irradiation in an earlier study employing the same laser conditions.[1] The null hypotheses in this study were that there was no difference in tensile bond strength between mechanically uniformly irradiated and free-hand irradiated dentin, and that laser setting had no effect on tensile bond strength in mechanically uniformly irradiated dentin.

MATERIALS AND METHODS

Laser Device and Group Setting

This study used a prototype Er:YAG laser device (J. Morita; Kyoto, Japan) set to emit a wavelength of 2.94 µm. The output energy of this device can be adjusted within a range of 30 to 250 mJ, and the pulse repetition rate can be adjusted within a range of 1 to 30 pulses per second (pps). The total energy delivered by the end of the probe, however, can only reach about 1.2 W. The pulse duration of this laser was set at approximately 400 µs. The teeth were divided into 3 lased groups, each subjected to a different output energy and pulse frequency (see Table 1). The total energy of each lased group was approximately 1.0 W. A 600-µm diameter straight-type contact probe was used. The energy levels were measured periodically with a power meter (Lasermate-P, Coherent; Santa Clara, CA, USA).

Specimen Preparation

Twenty-four bovine teeth were extracted, frozen to maintain freshness, then defrosted and cut at the cervix immediately before use in the experiments. The coronal sides of the cut surfaces were sequentially abraded under a stream of water with SiC paper (180-, 400-, and 600-grit) to prepare flat dentin surfaces. Next, they were randomly divided into 3 groups of 8 teeth each. The ground dentin surfaces were irradiated uniformly with an Er:YAG laser under a fine water spray. The tip of the laser was placed in light contact with the dentin surface to allow free movement, and the sample was fixed to an X-Y table (D-212, Suruga Seiki; Shizuoka, Japan) set to travel at a scanning speed of 1.0 mm/s. The dentin surfaces in each group were then conditioned with 10 wt% citric acid solution containing 3 wt% ferric chloride (Green activator, Sun Medical; Moriyama, Japan; batch no. ES1) for 15 s, rinsed with distilled water for 30 s, sufficiently dried, and bonded to square PMMA rods (8.0 x 8.0 x 8.0 mm) using 4-META/MMA-TBB resin (Superbond C&B, Sun Medical; C&B Metabond, Parkell; Farmingdale, NY, USA) by the brush-dip method. The batch numbers of this resin were GK2 (Polymer; Clear), KG2 (Monomer), and KE43 (TBB-O catalyst). The specimens were then subjected to the tests and observations detailed below.

Tensile Bond Testing

The specimens were kept at room temperature for 60 min immediately after preparation and then stored for 24 h in water maintained at 37°C. The bonded teeth were then serially sectioned vertically using a low-speed diamond saw (Isomet, Buehler; Lake Bluff, IL, USA) to create 2.0-mm-thick bonded dentin slabs. Each bonded slab was trimmed using a diamond point (FG #211 Regular, Shofu; Kyoto, Japan) high-speed air turbine handpiece under copious air-water spray to create a mini-dumbbell-shaped test specimen with a 3.0- x 2.0-mm cross section at the adhesive interface. After fixing the prepared specimens to a disposable PMMA jig, tensile strength was measured using a universal testing machine (Tensilon RTC-1150-TSD; Orientec; Tokyo, Japan) at a crosshead speed of 0.5 mm/min.

Mean tensile bond strength was evaluated by one-way and two-way ANOVA followed by the Fisher's PLSD test at a 95% level of confidence using StatView 5.0J (SAS institute; Berkeley, CA, USA).

SEM Observation of Fractured Surface

After tensile bond testing, each fractured specimen was placed on an aluminum stub and coated with Au-Pd using a Cool Sputter Coater (SC500A; VG Microtech; East Sussex, UK). They were then examined under a field-emission scanning electron microscope (SEM: ERA-8900FE, Elionix; Tokyo, Japan) to determine mode of failure.

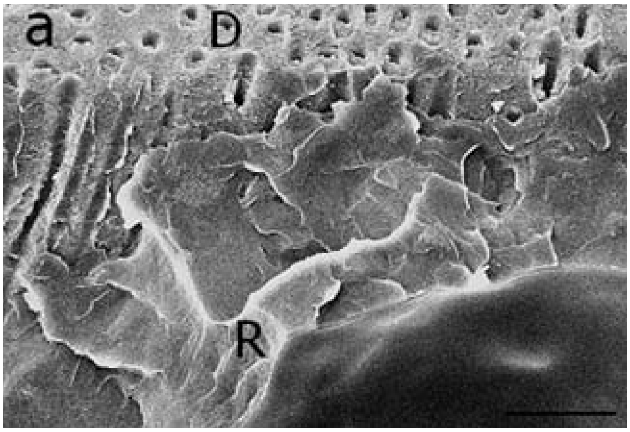

Fig 1a SEM micrograph of fractured dentin side after tensile bond strength testing (uniform irradiation at 100-10). Mixed failure mainly in non-resin-impregnated dentin (D) and cured resin (R). Bar = 20 µm.

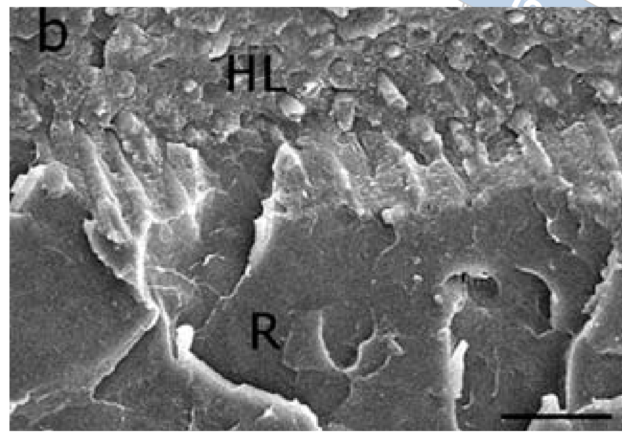

Fig 1b SEM micrograph of fractured rod side surfaces after tensile bond strength testing (uniform irradiation at 100-10).Mixed failure, mainly in cured resin (R) and partially hybrid layer (HL). Bar = 20 µm.

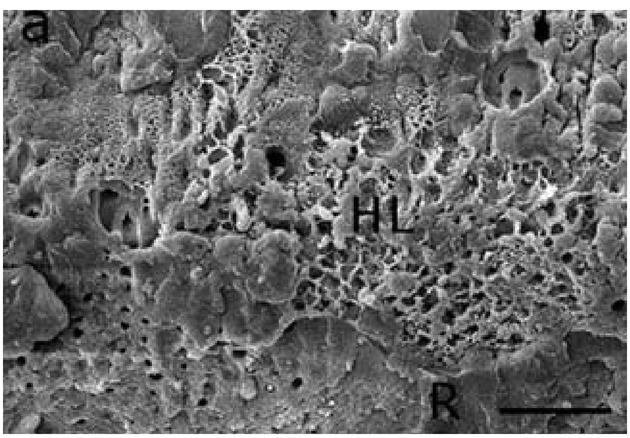

Fig 2a SEM micrograph of fractured dentin side after tensile bond strength testing (uniform irradiation at 50-20). Same as in 100-10 group, mixed failure revealed; however, area of cohesive failure in resin (R) was smaller than that in Fig 1. HL = hybrid layer. Bar = 20 µm.

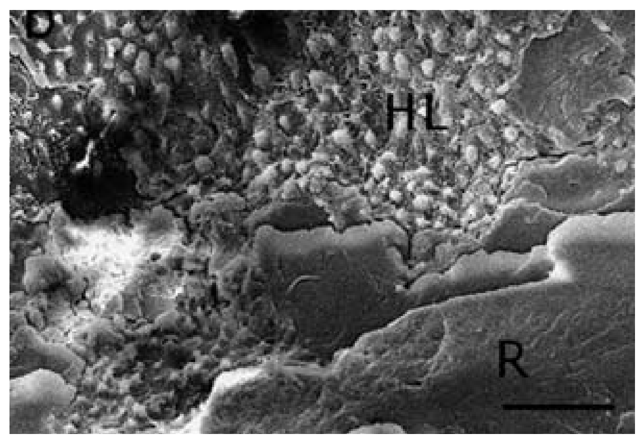

Fig 2b SEM micrograph of fractured rod side surface after tensile bond strength testing (uniform irradiation at 50-20). Same as in 100-10 group, mixed failure revealed; however, area of cohesive failure in resin (R) was smaller than that in Fig 1. HL = hybrid layer. Bar = 20 µm.

RESULTS

Tensile Bond Strength

Tensile bond strength and coefficient of variation (CV) in each group are shown in Table 1. The two-way ANOVA revealed a significant difference for “setting of laser” at $p < 0.05$, but no significant difference for “irradiation method” or interaction between these factors at $p > 0.05$. Bond strength in the 100-10 group was significantly higher than that in the 50-20 and 33-30 groups ($p < 0.05$) with both uniform and free-hand irradiation. However, no significant differences in bond strength were observed between the 50-20 and 33-30 groups ($p > 0.05$).

No significant difference was found between uniform irradiation and free-hand irradiation at each laser setting ($p > 0.05$). The coefficient of variation (standard deviation/mean x 100) was also calculated, but no difference was found.

Features of Fractured Surfaces

SEM observations of the fractured surfaces after tensile bond testing are shown in Fig 1. Cohesive failure in the cured resin was mainly observed in the 100-10 group, with partial failure within the hybrid layer and non-resin-impregnated dentin (Fig 1). In the 50-20 and 33-30 groups, failure in the cured resin was almost nonexistent, whereas failure within/at the bottom of the hybrid layer was extensive (Figs 2 and 3). When comparing the results of the previous[1] and present study, no differences were observed between free-hand and uniform irradiation at each laser setting.

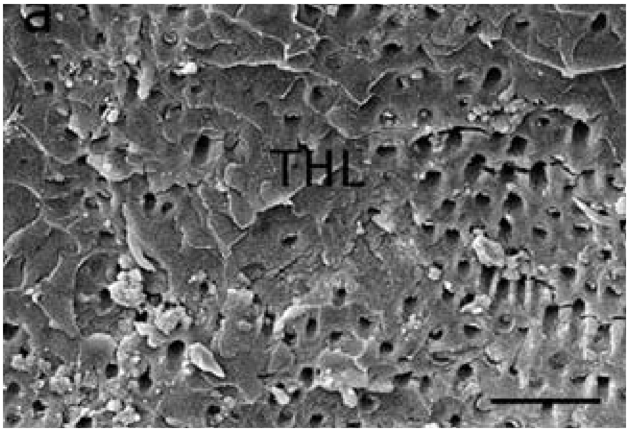

Fig 3a SEM micrograph of fractured dentin side surface after tensile bond strength testing (uniform irradiation at 33-30). Cohesive failure in resin almost nonexistent. Most failure occurred at interface between resin and top of hybrid layer (THL). Bar = 20 µm.

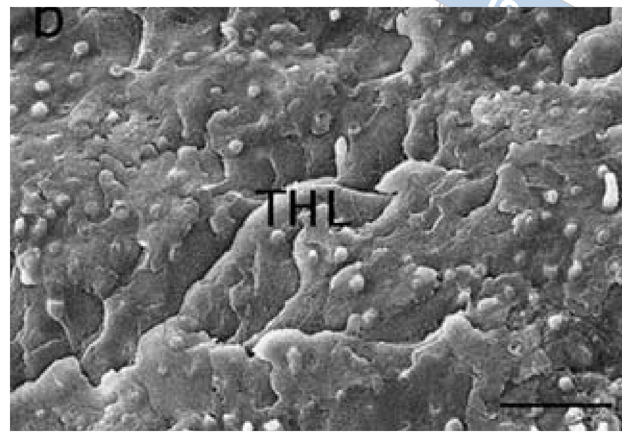

Fig 3b SEM micrograph of fractured rod side surface after tensile bond strength testing (uniform irradiation at 33-30). Cohesive failure in resin almost nonexistent. Most failure occurred at interface between resin and top of hybrid layer (THL). Bar = 20 µm.

DISCUSSION

In this study, we used 4-META/MMA-TBB resin as the adhesive system. This resin is not usually used in a clinical setting for Er:YAG laser-prepared cavities. However, this adhesive has often been used in the investigation of dentin bonding over the last 20 years. It has been used to study the effects of different acid conditioners,[15] additional application of monomer,[20,24,32] bond durability with fluoride-containing polymer,[30] and bond strength to heated dentin. Kameyama et al have also reported bond strength to Er:YAG laser-irradiated dentin with this resin, especially regarding the effect of different acid conditioners and the additional application of HEMA or glutaraldehyde.[17-19]

Several factors are believed to affect bond strength to lased dentin, including laser condition; experience with bonding procedures[25,31] and laser irradiation, individual dentin substrates,[29] and remaining dentin thickness.[7,33,37] In this study, all the specimens in the 6 groups were prepared by the same investigator (K.A.) to exclude the influence of operator experience. Furthermore, to exclude the factor of depth of bonded dentin, we used cross-sectioned labial dentin perpendicular to the tooth axis at the cervix, including superficial to deep dentin, as the bonded dentin surface.[15,24] A comparison of mechanical and free-hand irradiation under the same laser settings yielded no significant differences. No difference was found in the CV[3] between free-hand and mechanical irradiation at each setting. CV has been reported to be an indicator of consistency of bond strength;[3] therefore, these results allow us to reject the first null hypothesis.

The influence of output energy and pulse frequency on bond strength has been reported. Monghini et al[26] found no significant differences among specimens treated with 60 mJ-2 Hz, 80 mJ-2 Hz, and 100 mJ-2 Hz; however, an increase in laser output energy resulted in an increase in cratered surfaces, regardless of acid-etching association. Gonçalves et al[11,12] compared the influence of pulse frequency in both Er:YAG-lased enamel and dentin. While they found no significant differences among 80 mJ-1 Hz, 80 mJ-2 Hz, 80 mJ-3 Hz, and 80 mJ-4 Hz for enamel, increased frequency significantly decreased bond strength to dentin. This study compared bond strength to high-pulse frequency Er:YAG-lased dentin in 3 groups, in which the total energy used was adjusted to 1.0 W at the same moving speed. Among the 3 groups, the 100-10 group yielded significantly higher tensile bond strength than the 50-20 or 33-30 groups ($p < 0.05$), between which there was no significant difference ($p > 0.05$). These results also allow us to reject the second null hypothesis that laser setting had no effect on tensile bond strength. A difference was also found in fracture pattern after tensile bond testing among the 3 groups. In the 100-10 group, there was a mixture of failure, mainly in the cured resin and partially within the resin-impregnated or laser-modified dentin. On the other hand, the 50-20 and 33-30 groups showed failure mainly between the cured resin and the resin-impregnated or laser-modified dentin. These results suggest that the denatured dentin layer in the 100-10 group was of a different chemical or physical nature to that in the other two groups. The heat accumulation effect of the pulsed laser is determined by the relationship between pulse duration, pulse interval, and thermal relaxation time.[10,13,27] Irradiation applied at the same pulse duration with a high pulse frequency and lower pulse interval might easily lead to a greater accumulation of heat compared to irradiation at low pulse frequency. Physically speaking, pulsed laser irradiation at a high pulse repetition may easily denature tissue via heat accumulation, provided that the total energy is the same. Thus, the size of the denatured layer produced by the Er:YAG laser may depend on output energy, while the quality may depend on pulse frequency.

This study revealed that a high pulse frequency resulted in an adverse effect on bond strength in Er:YAG laser-irradiated dentin. Both the size and depth of the abrasions in the 33-30 and 50-20 groups were previously reported to be significantly smaller than those in the 100-10 group.[14] Al-though laser devices capable of laser irradiation at a high pulse frequency are now available, their effective use in a clinical setting remains problematic.

CONCLUSION

In conclusion, this study found no significant difference in tensile bond strength between free-hand and mechanically irradiated dentin. Significant differences were found, however, with change in laser setting in both the free-hand and mechanically irradiated groups. In particular, a high pulse frequency resulted in an adverse effect on bond strength in Er:YAG laser-irradiated dentin. Further study is needed to evaluate the usefulness of the high pulse frequency Er:YAG laser.

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Clinical relevance: A high pulse frequency resulted in an adverse effect on bond strength in Er:YAG laser-irradiated dentin.