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Resin Bonding to Dentin Irradiated by High Repetition Rate Er:YAG Laser

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ABSTRACT

Objective: This study evaluated the influence of laser irradiation with a high pulse repetition rate on dentin bonding. **Background Data:** Although resin bonding to erbium:yttrium-aluminum-garnet (Er:YAG) laser-irradiated dentin has frequently been investigated, the effects of a high pulse repetition rate have not yet been sufficiently investigated. **Methods:** Four groups treated under different laser conditions were evaluated in this study: 100 mJ/pulse–10 pulses per second [pps], 50 mJ/pulse–20 pps, 33 mJ/pulse–30 pps, and the unlased condition as a control. The total energy used to irradiate each group was adjusted to 1.0 W. After bovine dentin specimens were irradiated by an Er:YAG laser, acid conditioners (10% citric acid/3% ferric chloride) were applied to the lased surface. Thereafter, a PMMA rod was bonded to the lased dentin using 4-META/MMA-TBB resin, and mini-dumbbell-shaped specimens were prepared. These specimens were then tested under tensile mode and fractured surfaces were observed under scanning electron microscopy (SEM). **Results:** The bond strength of the unlased control was significantly higher than those of the three lased groups. Among the three lased groups, irradiation with higher output energy and lower pulse repetition rate tended to affect the higher bond strength. Upon SEM observation of the fractured surface, the lased groups showed the mixture of failure in the hybrid layer in almost part. There was no significant difference among the three lased groups. **Conclusion:** It can be concluded from the results of this study that a higher pulse repetition rate is not effective for resin bonding to laser-irradiated dentin.

INTRODUCTION

MANY INTERESTING SUGGESTIONS about resin bonding to erbium:yttrium-aluminum-garnet (Er:YAG) laser-irradiated enamel or dentin have been reported in recent years.^{1–17} Although almost all the research has indicated that laser irradiation has no or only a slight influence on resin bonding to enamel,^{1,3,5} there is some controversy concerning resin bond strength to dentin irradiated by Er:YAG laser. However, most researchers have reported that the resin bond strength to lased dentin was lower than that to unlased dentin.^{5–10,13–15} The reduction of dentin bonding strength has been discussed due to the denatured layer of dentin surface.^{16–20} Therefore, many researchers attempted to remove the denatured layer mechanically and chemically to improve the resin bond strength to

irradiated dentin.^{9,10,14} However, efficient methods for clinical application have not yet been established.

A prototype Er:YAG laser with high pulse repetition up to 30 pulses per second (pps) has recently been developed for efficient ablation.²¹ Some researchers have reported that a high pulse rate Er:YAG laser with low-energy output decreases the layer of denatured dentin.^{17,19} However, the influence of the laser pulse repetition rate on bonding strength has not been investigated to date. Therefore, we hypothesized that the high pulse repetition rate of Er:YAG laser would facilitate a higher resin bonding strength in comparison with a low pulse repetition rate.

This study evaluated the effect of high or low pulse rate Er:YAG laser on resin bonding strength to Er:YAG laser-irradiated dentin. The null hypothesis tested in this study was that

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any relationship was not found between laser conditions (output energy and pulse repetition rate) and bond strengths.

METHODS

Laser device and group setting

The laser equipment used in this study was a prototype Er:YAG laser (J. Morita Mfg. Corp., Kyoto, Japan) emitting a wave length 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 30 pps. However, the total energy at the end of probe is limited to approximately 1.2 W. In this study, three lased groups and an unlased control were established, as shown in Table 1. The total energy of each lased group was approximately 1.0 W. A 600- μm -diameter straight-type contact probe was used. Energy levels were measured periodically with a power meter (Lasermate-P, Coherent, CA).

Specimen preparation and tensile bond testing

Schematic illustrations of specimen preparations and tensile bond testing are shown in Figure 1. Thirty-two extracted bovine teeth, frozen to maintain freshness, were defrosted and cut at the cervix. The coronal sides of the cut surfaces were sequentially abraded under a stream of water with SiC paper of 180-, 400- and 600-grit to prepare flat dentin surfaces. Then the preparations were randomly divided into eight teeth for each group. The ground dentin surfaces were irradiated uniformly as possible with an Er:YAG laser using a fine water spray during operation by a free hand technique.

The dentin surfaces of each group were then conditioned with 10 wt% citric acid solution containing 3 wt% ferric chloride (Green activator, Sun Medical, Moriyama, Japan) for 15 sec, rinsed with distilled water for 30 sec, and sufficiently dried. Thereafter, these conditioned dentin surfaces were bonded to square PMMA rods (8.0 \times 8.0 \times 8.0 mm) using 4-META/MMA-TBB resin (Superbond C&B, Sun Medical Co., Moriyama, Japan; C&B Metabond in North America, Parkell) by the brush dip method. The samples were kept at room temperature for 60 min, then stored in 37°C water for 24 h.

The bonded teeth were then serially sectioned vertically to make 2.0-mm-thick bonded dentin slabs, using a low-speed diamond saw (Isomet™; Buehler, Lake Bluff, IL). Each bonded slab was trimmed to a miniaturized dumbbell-shaped test specimen^{22,23} with a 3.0 \times 2.0 mm cross-section at the adhesive interface using a diamond point (FG no. 211 regular; Shofu,

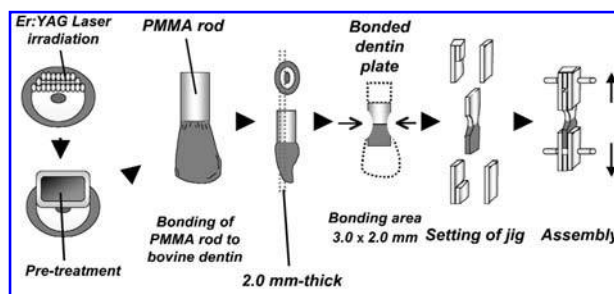


FIG. 1. Schematic illustration of the preparation of mini-dumbbell-shaped bonded specimens.

Kyoto, Japan) in a high-speed air turbine handpiece with copious air-water spray during operation as shown in Figure 2.

The prepared specimens were affixed to a disposable PMMA jig, and tensile strengths were measured using a universal testing machine (Tensilon RTC-1150-TSD; Orientec Co., Tokyo, Japan) at a cross-head speed of 0.5 mm/min.

Statistical analysis

There were eight samples in each group. The mean micro-tensile bond strength results were evaluated by one-way analysis of variance (ANOVA) followed by Fisher's PLSD test at the 95% level of confidence interval using StatView software.

Scanning electron microscopy observation of fractured surface

After tensile bond testing, each fractured specimen was placed on an aluminum stub, and Au-Pd coated using a Cool Sputter Coater (SC500A; VG Microtech, East Sussex, UK). The coated specimens were examined under SEM (SEM; JSM-6340F; JEOL, Tokyo, Japan) to determine the mode of failure.

RESULTS

The tensile bond strengths of each group are shown in Table 2. ANOVA demonstrated significant differences among the four investigated groups ($p < 0.0001$). The bond strength of the unlased control was significantly higher than that in any of the three lased groups. Among the three lased groups, irradiation with higher output energy and lower pulse repetition rate tended to affect the higher bond strength. However,

TABLE 1. GROUP SETTING IN THIS STUDY

Group	Output energy	repetition rate	Total energy
A	100 mJ/pulse	10 pps	1.0 W
B	50 mJ/pulse	20 pps	1.0 W
C	33 mJ/pulse	30 pps	1.0 W
D	Unlased control		0 W

^aMean values designated with the same letter are not significantly different ($p > 0.05$).
N = 8

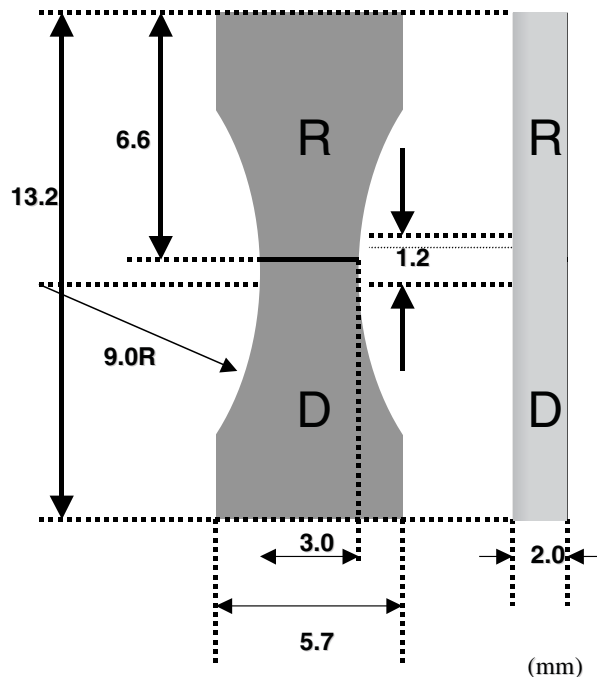


FIG. 2. Schematic illustration of the preparation of the sizes of mini-dumbbell-shaped bonded specimens. R, PMMA rod; D, bovine dentin.

there was no significant difference between Groups B and C ($p = 0.7274$).

The SEM views of fractured surfaces after the tensile bond testing are shown in Figure 3, and the failure modes of the fracture surfaces in each group are summarized in Table 2. In the unlasd control, almost the entire specimen showed cohesive failure in cured resin and adhesive failure was observed in parts (Fig. 3d). However, the lased groups showed the mixture of failure in the top and within the hybrid layer across almost the entire specimen (Fig. 3a–c). There were no significant differences among the three lased groups.

DISCUSSION

Laser irradiation with lower output energy has been reported to decrease pulpal damage occurring under the same pulse rep-

etition rate.²⁴ Lower output energy has also been reported to reduce the morphological changes in the lased surface.^{21,25} Generally, the removal of dentin caries by Er:YAG laser has been performed at ~60–100 mJ/pulse, ~1–10 pps (Hz).^{26–29} Recently, a new irradiation method using a lower output energy and higher pulse repetition rate has been developed for both lower pulpal damage and improvement of ablation efficiency.²¹ Ihara et al.¹⁹ reported that dentin ablation with low-power/high-pulse rate Er:YAG laser irradiation decreased the thickness of the laser-damaged dentin layer.

In this study, we compared bond strengths to Er:YAG laser-irradiated dentin in three lased groups in which the total energy used to irradiate each group was adjusted to 1.0 W, and an unlasd control (Table 1). It was found that the bond strength of the unlasd control (Group D) was significantly higher than those of the three lased groups. Therefore, we found that lased dentin decreased the resin bond strength in comparison with unlasd dentin. These results confirmed again the findings of many previous reports^{5–9,11–15} that laser-irradiation of dentin decreased bond strength. The fracture mode in Groups B and C after tensile bond testing mainly involved the top/within the hybrid layer, and partially involved the cured resin. In Group A, the area of the fracture in the cured resin was slightly larger than that in either Group B or C. The unlasd control showed a cohesive failure in cured resin throughout. In short, high bond strength demonstrated a cohesive failure in cured resin, whereas low bond strength was demonstrated as a failure in the top/within the hybrid layer. Each fracture mode corresponded with the results of resin bond strength test.

Mini-dumbbell bonded specimens, as recommended by Nakabayashi and Arao,^{22,23} were prepared for tensile bond testing. This method was reported to easily and exactly demonstrate the weakest region in the bonded specimen.²³ The SEM observations suggested that the resin impregnated layer composed of cured resin and laser-affected dentin was the weakest region on bonding to laser-irradiated dentin. It is well known that there are morphological changes in the lased surface after Er:YAG laser irradiation. Polarizing microscopic and light microscopic evaluations demonstrated a maximum of 600 μm depth of discolored area.¹⁶ Ceballos et al.¹¹ observed the adhesive interface between Er:YAG laser-irradiated dentin and restored resin composite by transmitted electron microscope and reported the denaturing/fusing of dentin components. Ishizaka et al.¹⁶ have been reported that Er:YAG laser-irradiated dentin could not distinguish the absorption band of C-H, C=O (amide I), and C-N (amide II) with Fourier-transformed spectrometry (FT-IR) analysis, although there was no significant difference

TABLE 2. TENSILE BOND STRENGTHS OF EACH GROUP AND SUMMARY OF THE FAILURE MODE

Group	Output energy	Repetition rate	Mean \pm SD (MPa)	Statistics ^a	Failure mode
A	100 mJ/pulse	10 pps	16.6 \pm 4.4	a	R > HL > D
B	50 mJ/pulse	20 pps	10.7 \pm 5.1	b	R > HL > D
C	33 mJ/pulse	30 pps	9.9 \pm 1.6	b	HL > R > D
D	Unlasd control		22.9 \pm 5.7	c	R > A

^aMean values designated with the same letter are not significantly different ($p > 0.05$). $N = 8$.

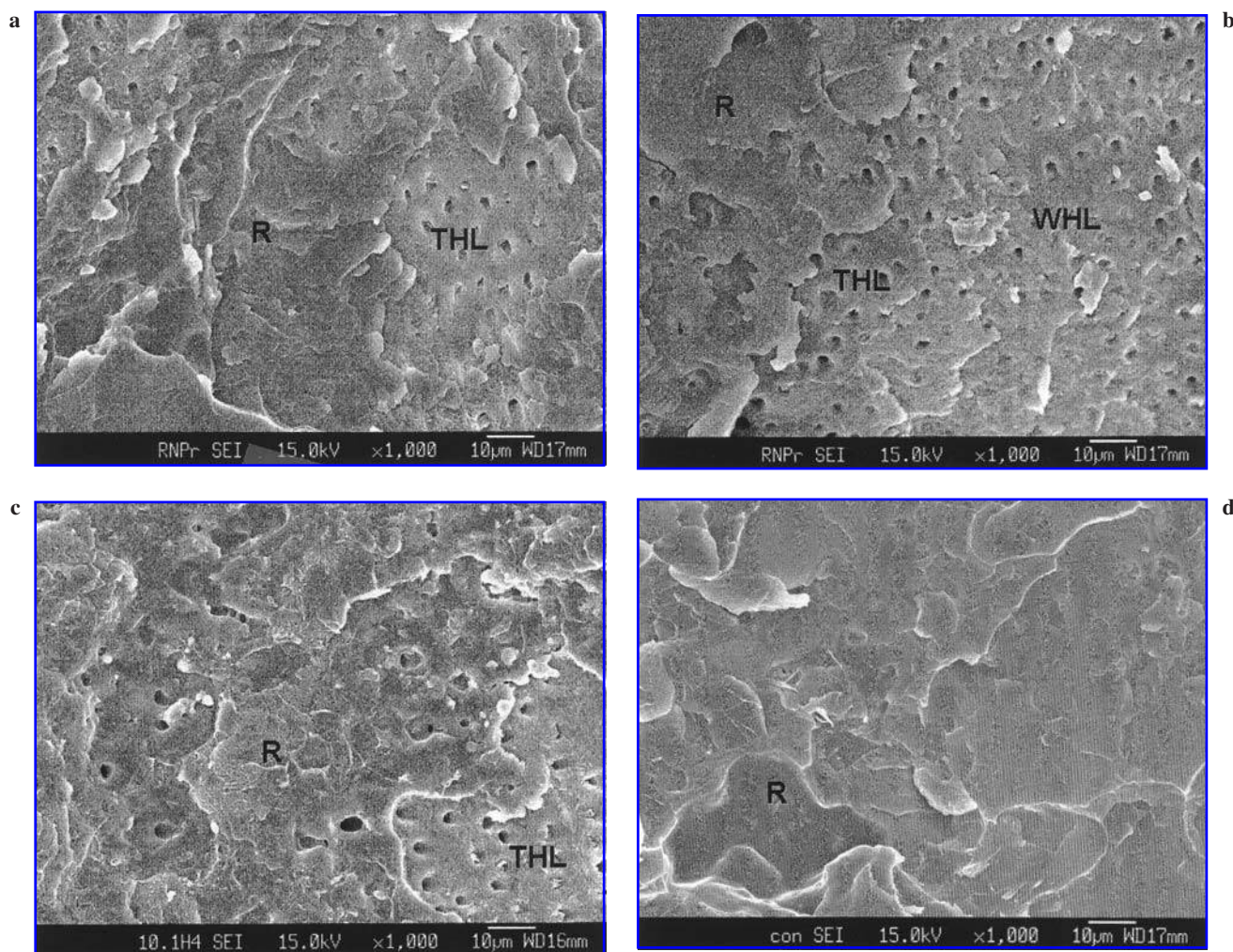


FIG. 3. Scanning electron microscopy (SEM) micrograph of the fractured dentin-side surface of a bonded specimen. (a) 100 mJ/pulse, 10 pps (15.3 MPa). (b) 50 mJ/pulse, 20 pps (11.2 MPa). (c) 33 mJ/pulse, 30 pps (8.1 MPa). (d) unlased control (25.4 MPa). Mixed failure of cohesion in cured resin (R), in the top of hybrid layer (THL), and within the hybrid layer (WHL) was seen in the lased three groups (a–c). Almost all of the specimen showed cohesive failure in cured resin (R) and adhesive failure (A) was partially seen in unlased control (d). Original magnification, $\times 1000$.

in the x-ray absorption on x-ray microradiographic evaluation. Iwata²³ also reported the percentage of hydroxyproline in lased dentin collagen was lower than that of unlased dentin collagen, although there was no significant difference on the x-ray diffraction analysis. Their findings indicated that there was no difference in crystal substances between acid-etched dentin and laser-irradiated/acid etched dentin, but there was a marked difference in the denaturation of organic materials in dentin. Moreover, Er:YAG laser irradiation with low output energy has been reported to decrease the depth of the laser-affected layer.¹⁹

In this study, we hypothesized that the high pulse repetition rate of Er:YAG laser would be able to achieve a higher resin bonding strength in comparison to that with a low pulse repetition rate. However, our results showed that the resin bonding strength achieved with a low pulse repetition rate using a Er:YAG laser and high energy was significantly higher than that achieved with a high pulse repetition rate and low energy.

Therefore, the hypothesis investigated in this study was rejected. From SEM observation of fracture surface, it was demonstrated that the area of the fracture within the hybrid layer after treatment with a high pulse repetition rate with low energy was larger than that of low pulse repetition rate and high energy. Therefore, the resin bonding strength does not depend on the thickness of the denatured layer but on the mechanical quality of the denatured layer. A high pulse repetition rate seemed to decrease the mechanical qualities of the denatured layer. Further studies should investigate the effect of a combination of pulse repetition rate and output energy on the strength of resin bonding to Er:YAG laser-irradiated dentin.

CONCLUSION

It can be concluded from the results of this study that (1) the bond strength to Er:YAG laser-irradiated dentin with a

high pulse repetition rate was significantly lower than that of unlased dentin; and (2) at an identical total energy of laser irradiation, a higher pulse repetition rate decreased bond strength.

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