Influence of an Optically Thick Water Layer on the Bond-Strength of Composite Resin to Dental Enamel After IR Laser Ablation

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Background and Objectives: Several studies of hard tissue ablation with Er:YAG lasers have shown that the addition of an optically thick water layer (~1 mm) added to the surface of dental enamel before each incident laser pulse, profoundly influences the rate and efficiency of ablation and the resulting surface morphology. The objective of this study was the determination of laser parameters which result in clinically useful bond strengths without the need for phosphoric acid etching. The hypothesis to be tested was that laser irradiation through a relatively thick layer of water would result in a surface to which composite could be bonded with bond strength similar to surfaces etched with phosphoric acid. This hypothesis is predicated on the assumption that the water prevents the formation of non-apatite calcium phosphate phases on the enamel surface.
Materials and Methods: In this study, a calibrated syringe pump and a motion control system were used to uniformly treat flat enamel surfaces using free-running Er:YAG laser pulses with and without water, and 9.6 μm CO_2 laser pulses on a dry surface for comparison. The rate of water delivery that resulted in the most efficient ablation was determined by profiling the resulting laser incisions using optical coherence tomography. In addition, enamel surfaces of 5×5 mm^2 were uniformly treated and the resulting surface morphology was examined using synchrotron radiation-fourier transform infrared spectroscopy (SR-FTIR), and optical and electron microscopy. The influence of the modified surface morphology on the adhesion of composite resin was investigated.
Results: The shear-bond strength of composite bonded to enamel surfaces irradiated at intensities clinically relevant for caries removal approached values measured for conventional acid etching when the water delivery rate was optimized.
Conclusions: This study demonstrates that composite restorative materials can be directly bonded to laser prepared surfaces without the necessity of further surface preparation and acid etching and that the addition of a thick water layer (~1 mm) prevents the formation of undesirable CaP phases that compromise adhesion to restorative materials. Lasers Surg. Med. 33:264–269, 2003.

Key words: CO_2 laser; dental enamel; Er:YAG laser; laser ablation; shear-bond strength; water augmentation

INTRODUCTION

Previous studies have shown that the Er:YAG laser ablation rate and efficiency for enamel removal can be increased by applying a water spray or a static layer of water to the surface before ablation [1–7]. Moreover, the addition of water has been found to alter the surface morphology and chemical composition of the irradiated enamel [8–10]. Such changes are likely to significantly modify the resistance of the laser irradiated enamel surface to acid dissolution and reduce the adhesion to restorative materials. Our previous investigation of the mechanism of water augmentation suggested that the forces imparted to the enamel surface by recoiling water during cavitation remove undesirable calcium phosphate phases and any surface protrusions or asperities in the ablation crater that are likely to inhibit efficient ablation of subsequent laser pulses leading to stalling and excessive heat accumulation [10].

Several studies have shown that the resin–enamel bond strength of Er:YAG laser treated surfaces to composite is highly variable and is typically less then that attained by conventional acid etching [11–18]. Moreover, other studies have shown that if mechanical means are employed to remove the loosely attached, poorly crystalline fused enamel particles and surface asperities, higher bond strengths are achievable [15]. In addition, Altshuler et al. [4] have shown that particle bombardment during laser ablation with either silica or hydroxyapatite particles can markedly improve the crater surface morphology. Since previous studies have demonstrated that a water layer of suitable thickness aids in the removal of such undesirable phases we hypothesized that we could optimize the laser–water interactions to produce a more suitable laser treated enamel surface for bonding to composite. It is important to note that the laser irradiation intensities employed in this

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study were commensurate with the optimum laser parameters for the most efficient removal of enamel, i.e., 25–100 J/cm^2. In contrast, in most previous studies, laser irradiation intensities were used that only roughen the enamel surface, and were markedly lower than those needed for clinical caries removal.

Majaron et al. [19] found that if the fiber was placed in a water droplet, there was a critical distance at which the enamel ablation was optimal. If the fiber to tooth distance was too small the ablation rate was low, most probably due to the failure to remove the modified enamel. If the fiber to tooth distance was too large, the laser pulse could not penetrate the water layer and the ablation rate was reduced significantly. Studies have shown that simple optical attenuation, as described by the Beer–Lambert law, does not apply here and the laser pulses penetrate through cavitation or parting of the water layer. Thus single Er:YAG laser pulses can penetrate through several millimeters of water even though the (1/e) absorption depth is calculated to be on the order of only 1 μm at 2.94 μm [20–22].

In this study, we first determined the optimum enamel ablation parameters for a free-running Er:YAG laser, and a long-pulse transverse excited atmospheric pressure (TEA) CO_2 laser operating at 9.6 μm, i.e., the irradiation intensity or fluence, spot size, scan distance, and the water delivery rate. After determination of the optimum conditions for water-mediated ablation, the surfaces of 5×5 mm^2 bovine enamel blocks were uniformly irradiated and the shear-bond strength to composite was measured using a mechanical testing apparatus.

MATERIALS AND METHODS

Laser Parameters

Blocks of bovine enamel were irradiated using a free-running Er:YAG (2.94 μm) laser system and a 9.6 μm long-pulsed CO_2 TEA laser (Argus Photonics Group, Jupiter, FL) with a pulse duration of 5–8 microsecond. The solid-state laser system was manufactured by Schwartz Electronics, FL (Schwartz 123 laser) and was modified for diffuse pumping. The laser energy was measured and calibrated using a laser calorimeter (Model ED-200-Gentec, Quebec, Canada). The beam diameter at the position of irradiation was measured by scanning with a razor blade across the beam. An inter-cavity aperture was used to reduce the output to a single-transverse mode and fluences were defined using a Gaussian beam with a 1/e^2 beam diameter.

Tissue Irradiation

Longitudinal cuts approximately 3 mm long and 200–300 μm wide were produced on the enamel surface of 5×5 mm^2 blocks from extracted bovine incisors, polished to a 1 μm finish. Incisions were made at varying fluences from 10 J/cm^2 to 100 J/cm^2 and varying water delivery rates from 0 ml/min to 2 ml/min using the Er:YAG laser. The laser spots were overlapping, separated by 50 μm to ensure a smooth intensity profile longitudinally through the incision. The procedure is illustrated in Figure 1. The bovine block was scanned across the laser beam using a computer

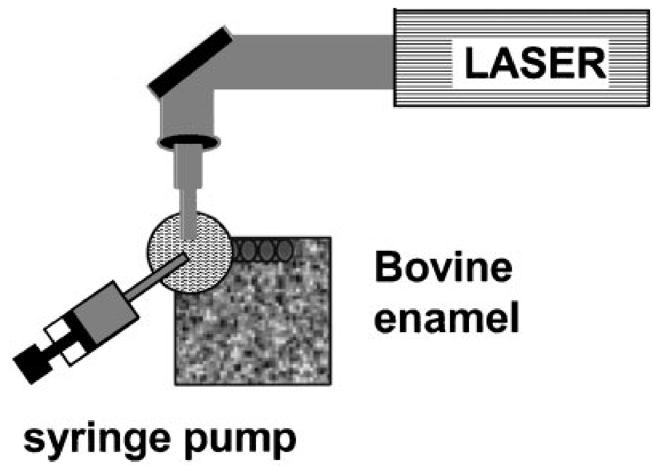

Fig. 1. The setup for creating lateral cuts in polished bovine enamel blocks. A syringe was used to manually apply a droplet of water before each of the five laser pulses.

controlled motion control system ESP-300 (Newport, Irvine, CA) with an X–Y stage. Droplets of water were applied to the ablation site before each sequence of five laser pulses using a calibrated syringe pump (Model 100, KD Scientific, New Hope, PA) with a resolution of 0.1 ml/hr. The water pooled into a drop encompassing the surface of the bovine block with a thickness exceeding 1 mm. Incident laser pulses removed the water layer and the rate of replenishment was determined by the rate of delivery by the syringe pump. After surface ablation, an Olympus microscope with a maximum magnification of 500-times, interfaced to a digital CCD camera, and image analysis software was used to acquire images of the ablated enamel. The depth of the lateral incisions was measured using a polarization sensitive-optical coherence tomography (PS-OCT) system (Fig. 2). The PS-OCT system was operated at 1,310 nm and had axial (depth) and transverse resolutions of 30 μm (1/e^2), respectively, the apparatus has been described elsewhere [23].

Synchrotron Radiation-Fourier Transform Infrared Spectroscopy (SR-FTIR)

A Nicolet Magna 760 FTIR interfaced to a Nic-Plan IR microscope, equipped with a motorized sample stage connected to Beam-Line 1.4.3 of the Advanced Light Source at Lawrence Berkeley National Laboratory was used to acquire spectra of the dental enamel irradiated with the CO_2 laser [24]. Specular reflectance spectra were acquired with a spatial resolution of 10 μm by scanning the 10 μm spot imaged by the FTIR microscope across the area of interest.

Shear-Bond Strength Measurements

Nine experimental sample groups ($n = 10$ blocks) were evaluated, including an non-irradiated positive control group that was acid etched according to the manufacturer's protocol, a non-irradiated negative control group without acid etch, and seven laser groups irradiated with and

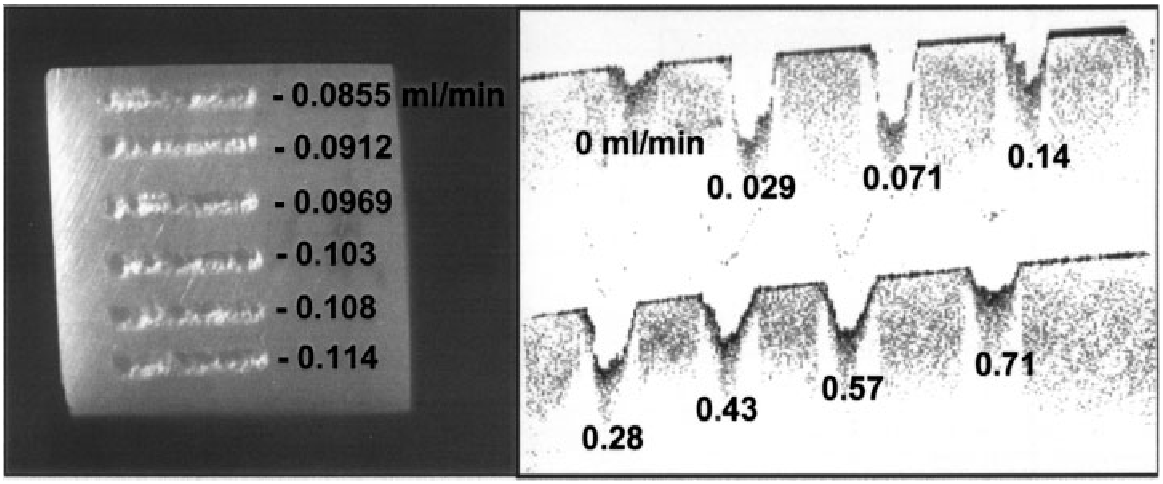

Fig. 2. **Left**: Incisions produced in the surface of a bovine enamel block for different water flow rates (ml/min) with a free-running Er:YAG laser pulses of 150 microsecond duration with an incident fluence of 100 J/cm^2 with a spot diameter of 300 μm a scan distance between spots of 150 μm, a pulse repetition rate of 3 Hz and six laser pulses per spot. **Right**: Optical cross sections of incisions produced at various water flow rates with the same laser irradiation parameters taken using optical coherence tomography.

without an optimized water application rate. An additional fourth laser group, the 9.6 μm CO_2 laser without water was added for comparison. The 9.6 μm CO_2 laser produces a well-defined melt layer of modified enamel on the surface [10]. The laser beam was focused to a spot diameter of ~300 μm and the bovine blocks were scanned across the laser beam to uniformly cover the entire 5×5 mm^2 surface of each bovine block with overlapping laser spots using a ESP-300 motion control system incorporating two 850F stages (Newport). Three or six pulses were delivered for each spot with a scan distance of 50, 100, or 150 μm between spots and a repetition rate of 3 Hz was used for each sample.

In order to access the suitability of laser treated surfaces for bonding, the shear-bond test was employed to evaluate the adhesive strength. Single bond and Z-250 composite (3M, Minneapolis, MN) were applied to the circular testing area (3.2 mm Ø) according to manufacturer's instructions. The non-irradiated positive control was etched with 35% phosphoric acid according to the manufacturer's recommendations, rinsed thoroughly with a water stream, gently dried, leaving a shiny moist surface, then the bonding resin was applied in two coats and dried, followed by a 10 second light cure. The negative control was neither irradiated by the laser nor acid etched, but simply rinsed thoroughly with water and gently dried prior to application of the bonding resin. The laser treated groups were not acid etched after laser irradiation but simply rinsed thoroughly with water and gently dried prior to application of the bonding resin. The modified single plane shear test assembly (SPSTA) was used according to the protocol developed by Watanabe et al. [25] in order to avoid the problems associated with shear tests using shearing knives or wire loops that produce shear–peel forces and not true shear forces. The SPSTA was attached to an Instron testing machine with two aligning plates. The Instron was calibrated and set to record measurements in kilograms. A crosshead speed of 5 mm/min was used. The force level (measured in kilograms) was recorded for each sample at the precise point when the two shear plates separated from each other. The recorded force-failure measurements were divided by the surface area of bonded region and multiplied by the conversion factor to convert the stress required for failure from kg/cm^2 to MegaPascals (MPa).

RESULTS

Optimization of Water Delivery Rate

Lateral cuts were produced in the surface enamel of the bovine blocks using the free-running erbium laser pulses. The craters were typically quite shallow when water was not used and areas of fused enamel were clearly visible. The incisions were made at an incident fluence of 100 J/cm^2, and the water delivery rate was varied from 0 ml/min to 2 ml/min. A representative series of incisions is shown in Figure 2 for water delivery rates of 0.08–0.12 ml/min. The depth of each incision was measured via transverse scans with optical coherence tomography. A series of scans is shown in Figure 2 for water delivery rates of 0–0.7 ml/min. The deepest trenches were produced for a water delivery rate of 0.09 ml/min.

Uniform Irradiation of Enamel Surfaces

After the optimum water delivery rate was determined to be 0.09 ml/min, the entire surface of the bovine enamel blocks were irradiated with and without water. The CO_2 laser was used without water at 10 J/cm^2. Initial Er:YAG samples were irradiated at a fluence of 100 J/cm^2 with a spot diameter of 300 μm, a scan distance between spots of 150 μm, a pulse repetition rate of 3 Hz, and six laser pulses per spot. The irradiated surfaces are shown at two magnifications in Figure 3. The high incident fluence coupled with the Gaussian laser profile and relatively large

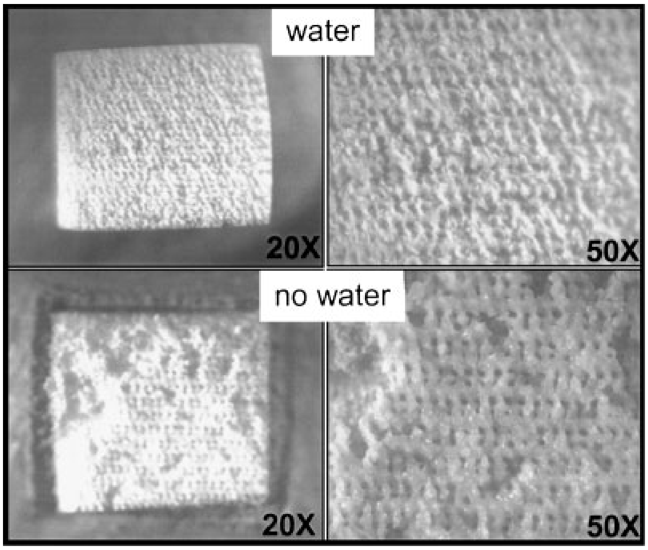

Fig. 3. Optical microscopic images at two magnifications of the surfaces of bovine enamel irradiated with free-running Er:YAG laser pulses of 150 microsecond duration with an incident fluence of 100 J/cm^2 with a spot diameter of 300 μm a scan distance between spots of 150 μm, a pulse repetition rate of 3 Hz and six laser pulses per spot. The two top images were treated at a water flow rate of 0.09 ml/min.

separation between individual laser spots produced a non-uniform surface with a pattern of small holes drilled into the surface. Therefore, the distance between laser spots was reduced from 150 μm ($\frac{1}{2}$ the spot diameter) to 50 μm ($\frac{1}{6}$ of the spot diameter). In addition, the number of laser pulses per spot was reduced to three and the incident fluence was reduced to 50 J/cm^2 to produce more uniform surface topography (Fig. 4). Surfaces irradiated without water contained large amounts of fused, i.e., melted and re-crystallized mineral phases. One sample, out of the 10 samples treated without water, had an intact surface layer of fused enamel that almost encompassed the entire surface. This particular sample is shown in the left image of Figure 4. The distribution of the deposits of fused enamel was not as uniform for the other nine samples

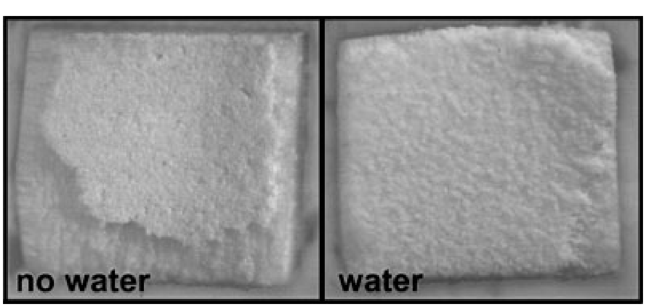

Fig. 4. Optical microscopic images at 20× magnification of the surfaces of bovine enamel irradiated with free-running Er:YAG laser pulses of 150 microsecond duration with an incident fluence of 50 J/cm^2, a spot diameter of 300 μm, a scan distance between spots of 50 μm, a pulse repetition rate of 3 Hz and three laser pulses per spot. The enamel block on the right was treated at a water flow rate of 0.09 ml/min.

Infrared Spectromicroscopy (SR-FTIR)

High resolution IR spectromicroscopy using a FTIR coupled to the high brightness ALS was used to acquire spectra from the Er:YAG sample irradiated without water with the intact layer of modified enamel, Figure 5. In the region of the sample shown in Figures 4 and 5 where the fused layer of enamel had exfoliated (light gray trace) and on the surfaces of the samples irradiated with water, the IR spectra resembles that of hydroxyapatite. In the region of the intact fused surface layer of Figure 5 there are changes in the phosphate peaks between 1,000 cm^{-1} and 2,000 cm^{-1} indicating changes in either the apatite crystal orientation or the appearance of other CaP mineral phases.

Shear-Bond Strength to Composite

The laser parameters and mean shear-bond strengths ± the standard deviation are shown in the Table 1. Additional Er:YAG laser parameters were a spot diameter of 300 μm, a pulse repetition rate of 3 Hz, three laser pulses per spot, and a water flow rate of 0.09 ml/min. A one-way ANOVA test showed that the means of the treatment groups are different with $P < 0.0001$. Tukey–Kramer multiple comparisons test provided the statistical groupings in the table with the significance level at $P < 0.05$. One of the Er:YAG laser treated groups was not statistically different from the positive control, and all of the laser treated groups were different from the negative control.

The de-bonded surfaces were examined for site of failure under a stereo microscope at a magnification of 10×. All the laser treated samples showed a mixed mode of failure, with

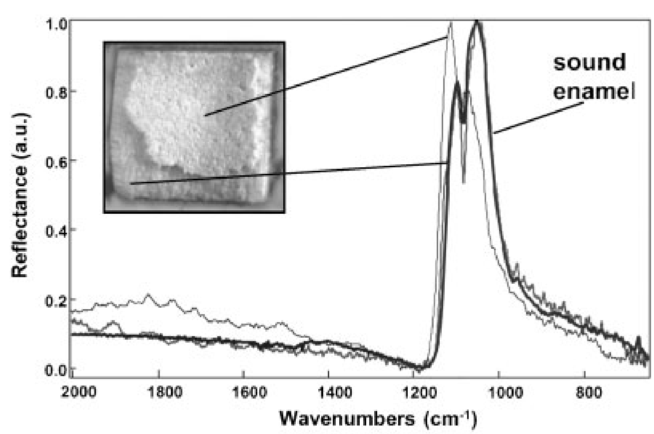

Fig. 5. Synchrotron radiation-fourier transform infrared spectroscopy (SR-FTIR) spectra taken of areas on the enamel block shown in the left image of Figure 4 treated without water. The thin black line represents the fused surface layer of the enamel, the thick black line represents sound enamel and the thick gray line represents the enamel underneath the layer of exfoliated enamel.

TABLE 1. Bond Strengths of Composite to Laser Treated Dentin

Surface treatment	Fluence (J/cm^2)	Scan distance (μm)	Water with laser	Mean ± SD (N)	Statistical grouping
Etch H_3PO_4	—	—		31.03 ± 5.26 (10)	a
Er:YAG laser	50	50	+	25.88 ± 3.58 (11)	a, b
Er:YAG laser	50	100	+	21.22 ± 4.64 (11)	b, c
Er:YAG laser	100	150	+	20.91 ± 5.35 (10)	b, c, d
CO_2 laser	10	100	−	18.52 ± 4.23 (9)	b, c, d, e
Er:YAG laser	50	50	−	18.12 ± 5.03 (11)	c, d, e
Er:YAG laser	100	150	−	17.86 ± 2.68 (10)	c, d, e
Er:YAG laser	25	100	+	11.37 ± 9.88 (11)	e
No etch	—	—		2.13 ± 1.75 (12)	f

no clear pattern. Some failure of laser-irradiated enamel appeared in all samples, but was frequent especially in the samples lased without water. The laser treatment appeared to affect the enamel to a depth of $\frac{1}{4}$ to $\frac{1}{2}$ mm, resulting in a rough, chalky appearance.

DISCUSSION

This study demonstrates that an applied water layer of sufficient thickness has a profound effect on ablation rate, ablation efficiency, surface morphology of the crater walls, and the adhesion to restorative materials. As we stated in our earlier study [10], we postulate that the the forces associated with strong absorption of the laser radiation in the water followed by the subsequent recoil of the water removes any poorly attached non-apatite phases of modified enamel that may adversely affect the bond strength, in addition to reducing the rate and efficiency of ablation. The optimum water delivery rate—0.09 ml/min that was identified in this study is markedly lower than the rates of water delivery that is used with current Er:YAG and Er:YSGG lasers, and the high-speed dental hand piece. That rate was for a repetition rate of only 3 Hz and most probably the rate of water delivery at 30 Hz would still be less than 1 ml/min [26]. The ablation rate was not very sensitive to the water delivery rate and in the range of 0.03–0.5 ml/min, the depth of cut did not vary by over 20–30%, indicating that optimal rates of delivery can readily be achieved "in vivo."

It was critical that the surface was uniformly treated and it was necessary to reduce the center to center distance between adjacent laser spots to 50 μm ($\frac{1}{6}$ the beam diameter). If this was not done, the bond strength was lower due to the high surface roughness and weakened integrity of the remaining enamel between each of the holes (see Fig. 4). The roughness or spatial modulation of the surface is another possible source of the wide variation in the bond strengths reported for Er:YAG laser irradiated enamel in the literature.

The shear-bond strength of composite bonded to enamel surfaces irradiated at irradiation intensities clinically relevant for caries removal, 50–100 J/cm^2 approached values measured for conventional acid etching when the water delivery rate was optimized, with one group showing no statistical difference from the control. This study demonstrates that composite restorative materials can be directly bonded to laser prepared surfaces without the necessity of further surface preparation and acid etching. This also supports our hypothesis that the addition of a thick water layer (~1 mm) prevents the formation of undesirable CaP phases that compromise adhesion to restorative materials. It is likely, that the strong recoil forces imparted to the water during the interaction of the laser pulse with the thick water layer removes loosely attached CaP phases peripheral to the site of ablation, thus preventing the accumulation of these undesirable phases during multiple irradiation.

The current standard treatment for bonding resinous materials to enamel is still phosphoric acid etching. The bonding agents have evolved over the last two decades and more options have become available, with various combinations of primers and resins sometimes combined in "one-bottle" systems. Recently, self-etching primers have become popular for treating both enamel and dentin, because of fewer steps and less technique sensitivity. The phosphoric acid step is eliminated in these systems. On dentin, the bond strengths obtained with self-etching primers are usually as good or better than those obtained with etch and prime systems. However, on uncut enamel, the self-etching primers may not perform as well, because of the less aggressive etching produced by them [27]. In the present study, we used a "one-bottle" adhesive which combines the primer and resin in the same bottle. This system was chosen because it represents a popular approach to bonding and includes the phosphoric acid etching step that has been the gold standard for enamel bonding since the advent of adhesive dentistry. It is possible that other adhesives might yield different results and future research should explore the interaction of various laser treatments with other types of adhesives in producing a suitable bond.

Previous studies indicated a greater influence of the applied water layer on the ablation rate if shorter laser pulses were used, namely Q-switched (150 nanosecond) pulses [10]. Cleaner, smoother ablation craters were produced with water coupled with the shorter Er:YAG laser pulses. In future investigations, we will study the influence of the pulse duration on the optimal water delivery rate and the bond strength.

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