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Adhesion studies on dental enamel surfaces irradiated by a rapidly scanned carbon dioxide laser

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Abstract

In this study, we investigated the influence of different laser scanning patterns on the adhesive strength of laser irradiated enamel surfaces both with and without post ablation acid etching. Previous studies of dental enamel surfaces ablated by a rapidly scanned carbon dioxide laser indicated that the highly uniform smooth surfaces produced by the scanned laser beam yielded low bond strength and acid etching was required in order to attain a high bond strength. However, since the enamel surface after ablation by CO₂ lasers is more resistant to acid dissolution it is desirable to avoid acid etching before bonding. The overlap between adjacent laser spots was varied to modify the effective surface roughness. In addition, small retention holes were drilled at higher laser intensity with varying spacing to increase the adhesive strength without acid etching. Varying the degree of overlap between adjacent laser spots did not significantly influence the bond strength with post ablation acid etching. The bond strength was significantly higher without acid etching with retention holes spaced 250- μ m apart.

Keywords

enamel; CO₂ laser; adhesion; composite

1. INTRODUCTION

Several studies have demonstrated that carbon dioxide lasers operating at wavelengths of $\lambda = 9.3$ and $9.6\text{-}\mu\text{m}$ are ideally suited for the efficient ablation of dental caries and for surface treatments to increase the resistance to acid dissolution.¹⁻⁵ If pulse durations in the range of $5\text{--}20\text{-}\mu\text{s}$ are used efficient ablation can be achieved with minimal thermal peripheral damage^{3, 5, 6}. Transverse excited atmospheric pressure (TEA) CO₂ lasers operate through a simple high voltage discharge that excites the gas matrix and can potentially be manufactured at relatively low cost. Another advantage of this laser system is that it is capable of operating efficiently at high pulse repetition rates and the system can be combined with a laser beam scanning system for high-speed precision removal of dental caries. One concern at higher repetition rates is the increased potential for peripheral thermal damage due to heat accumulation from multiple laser pulses delivered in rapid succession. This heat accumulation can be offset by the rapid scanning of the laser beam. The residual heat deposition after laser ablation is an advantage for caries inhibition around caries

preparations but the thermally modified layer may reduce the bond strength to composite⁷⁻¹⁰. Last year we demonstrated that a rapid CO₂ laser scanning system operating with laser pulses of 10–15-μs and pulse repetition rates of 300 Hz operating at a wavelength of 9.3 μm could be used to rapidly remove ablated enamel while maintaining interpulpal temperatures at safe levels¹¹. Other studies showed that high shear bond strengths can be achieved on both enamel and dentin surfaces irradiated by this laser, however those bond strengths were significantly lower than the conventionally prepared surfaces that were acid-etched^{7-9,12}. If the enamel surfaces were not acid etched, they manifested low bond strength, ~ 6 MPa. This contradicted early studies in which we achieved markedly higher bond strengths for the laser irradiated surface even without water cooling, albeit the repetition rate was only 10 Hz in those studies. The TEA CO₂ laser was operated at a wavelength of 9.6 μm with a pulse duration of 5–8 μs, the spot size was 300-μm and the overlap between laser spots was 100-μm. The bovine enamel laser irradiated surface had a bond strength of 18.1 ± 4.2 vs. 2.1 ± 1.8 for the non-irradiated, non etched negative control group.¹² In a second study the higher bond strengths were confirmed both with and without water cooling at a higher fluence.⁹ Those surfaces appeared to be much rougher compared to the enamel surfaces treated at 300-Hz, therefore, we hypothesized that the higher adhesive strength could be caused by the increased surface roughness.

The surface topography can be easily manipulated using a computer controlled laser scanning system. The surface roughness or modulation can be increased by increasing the spacing or decreasing the spatial overlap between adjacent laser spots. Distinct patterns or retention holes can be rapidly drilled into the surface without decreasing the speed of ablation. The purpose of this study was to determine if the shear bond strength of enamel to composite could be increased by varying the laser scanning parameters to produce surfaces of varying roughness and surface topography.

2. MATERIALS AND METHODS

2.1 Samples

Noncarious, extracted teeth from patients in the San Francisco bay area were collected with approval from the UCSF Committee on Human Research, cleaned, sterilized with gamma radiation, and stored in a 0.1% thymol solution to preserve tissue hydration and to prevent bacterial growth. There were a total of 6 groups each containing at least 10 samples per group for repeated measurements. The samples were cut as shown in Fig. 1 to produce a flat surface with at least 4×4-mm of enamel using an Isomet 2000 Buehler (Lake Bluff, IL) precision saw and kept well hydrated before ablation. Samples were polished using 600 carbide grit, and the debris produced was removed by sonication.

2.2 Tissue Irradiation and Laser Parameters

An industrial marking laser, Impact 2500 from GSI Lumonics (Rugby, United Kingdom) operating at a wavelength of 9.3 μm was used. The laser was custom modified to produce a Gaussian output beam (single spatial mode) with a pulse duration of between 10–15-μs. This laser is capable of high repetition rates up to 500 Hz, and a fixed repetition rate of 300 Hz was used for these experiments. The laser energy output was monitored using a power/

energy meter, ED-200 from Gentec (Quebec, Canada). The laser beam was focused to a spot size of $\sim 450\text{-}\mu\text{m}$ using a planoconvex ZnSe lens of 125-mm focal length. The laser fluence was either 13 or 42 J/cm². A razor blade was scanned across the beam to determine the diameter ($1/e^2$) of the laser beam. Computer-controlled XY galvanometers 6200HM series with MicroMax Series 671 from Cambridge Technology, Inc. (Cambridge) were used to scan the laser beam over the sample surfaces. Figure 1 shows the basic setup for the laser apparatus. Following ablation, the blocks were embedded into the shear test jig with dental stone and tested using the shear-bond test.

2.3 Test Groups

Six test groups were used. These consisted of one non-irradiated etched group and five laser treated groups that produced surfaces of varying topography. The laser fluence was 13 J/cm², the spot size was 450- μm and the overlap between laser pulses was varied for three of the groups, Group L100-overlap 2/9 spot diameter:100- μm spacing, Group L200-overlap 4/9 spot diameter:200- μm spacing, and Group L450-overlap the spot diameter:450- μm spacing. These three groups and the control group were acid etched before bonding to composite. An array of small holes 100- μm deep were drilled in two of the groups to serve as retention holes, the surface was first uniformly irradiated using a 100- μm spacing between spots (L100) and then small holes were drilled separated by either 250- μm (Group R250) or 500- μm (Group R500) using a fluence of The laser fluence was 42 J/cm² with 20 laser pulses per spot. The two R-groups were not acid etched before bonding.

2.4 Shear-Bond Test

The adhesive strength of the bonding agent to enamel was determined by the shear-bond test. The bonding material was Single Bond along with the Z-250 composite, 3M-ESPE (Minneapolis, MN). The positive control group was only etched with 35% phosphoric acid, rinsed with water, and gently dried. The L-group of laser treated samples was also etched with 35% phosphoric acid, rinsed with water and gently dried. The other R-group of laser treated samples was not etched. Subsequently, the bonding resin was applied to all the blocks in two coats, dried, and cured for 20 seconds prior to bonding with composite.

The modified single plane shear test assembly (SPSTA) followed the procedure used by Sheth et al. and Watanabe et al.^{13, 14}. Figure 2 shows the shear-bond test setup for the SPSTA method. Two aligning plates were used to connect the SPSTA to an Instron testing machine, that recorded measurements in kilograms with the crosshead speed set to 5 mm/min. When the two plates separated, the force level was recorded. The force-failure data (in kilograms) was divided by the surface area of the region and a conversion factor was used to calculate the force in Mega-Pascals (MPa).

3. RESULTS

Minimal thermal damage was observed on all the laser treated surfaces. Figure 3 shows the surfaces of all the sample groups irradiated. The surface treated with the maximum overlap produced a very smooth uniform surface (L100) and none of the residual scratches from sample preparation that are visible in the controls samples can be seen. The shear bond

strengths for the six groups are shown graphically in Fig. 4. The specific values, mean $\pm$ standard deviation are: Control (36.6 ± 4.9 (n=12)), L100 (34.1 ± 4.6 (n=12)), L200 (34.9 ± 5.1 (n=12)), L450 (33.4 ± 5.5 (n=10)), R250 (15.3 ± 3.9 (n=10)) and R500 (5.8 ± 3.1 (n=10)). All the acid etched groups had bond strengths exceeding 30 MPa and even though the mean bond strength of the control group was higher than the laser treated sample groups they were statistically similar ($P > 0.05$). The sample group with the closely spaced retention holes (R250) had a high bond strength which was significantly higher than the holes spaced 500- μ m apart and significantly higher than the enamel samples prepared without retention holes (5.2 ± 2.4 MPa, n=10)¹¹ or the negative non-etched enamel control samples from previous studies (2.7 ± 2.3 MPa, n=12)⁹ and (2.1 ± 1.8 MPa, n=10)¹².

4. DISCUSSION

The enamel shear bond strength with post ablation acid etching was high and exceeded 30 MPa for all the laser groups. The difference in surface roughness between the three laser groups with varying spatial overlap did not appear to influence the bond strength. In a similar prior study involving the Er:YAG laser the surface roughness also did not markedly change the bond strength¹⁵. In our enamel adhesion study carried out last year with similar laser conditions the bond strength of laser treated enamel surface exceeded 30 MPa, (31.2 ± 2.5 MPa, n=8) but it was significantly lower than the control group which was 37 ± 3.6 MPa.

One surprise after our initial measurements last year was that the bond strengths after laser irradiation were extremely low if acid etching was not applied¹¹. This contradicts two previous measurements using a 9.6- μ m CO₂ laser with a slightly shorter laser pulse duration, 5–8 μ s that yielded fairly high bond strengths without the need for post ablation acid etching, namely 18.2 ± 7.4 MPa, n=10 with a water spray and 15.6 ± 4.3 , n=10 and 18.5 ± 4.2 , n=9 without a water spray^{9, 12}. One major difference between the more recent studies and the previous work is that the laser beam was scanned over the enamel surface producing a more highly uniform smooth surface. In previous studies the surface manifested a higher surface roughness that we assumed was responsible for the increased bond strength since no thermal damage is visible on the enamel surface. However, varying the scanning parameters in this study indicates that the difference in adhesive strength is likely not due to surface roughness. The repetition rate in the 9.6- μ m studies was low; only 10-Hz versus 300-Hz for this study, which can account for the difference. However, no thermal damage was apparent on the samples in this study and thermal damage should have been quite high in the two separate 9.6- μ m CO₂ laser studies in which water-cooling was not used, yet mean bond strengths of 15 and 18 MPa were achieved without acid etching^{9, 12}. Moreover, we see no difference between the bond strength without acid etching for dentin between the two repetition rates, even though dentin is more sensitive to thermal damage than enamel^{9, 12}. Additional studies at varying pulse repetition rates may be needed to resolve this issue. The post-ablation acid etched enamel surfaces have a slightly lower bond strength and it is not clear whether there is any clinical significance. It is also not clear whether the acid etch has completely removed the acid resistant modified enamel layer or that the laser-treated surfaces have a higher resistance to acid dissolution than the untreated enamel surfaces. There may be an optimum set of laser irradiation parameters and acid etching conditions that

can produce the desired surface roughness/morphology needed for adhesion while maintaining the increased resistance to acid dissolution.

In this study we also experimented with two groups in which small retention holes were deliberately produced between 100–200- μm deep and the surfaces were not etched prior to bonding. There was not much of an increase in bond strength for the holes spaced 500- μm apart but the bond strength increased by a factor of three to 15 MPa for the 250- μm holes which indicates that surface patterning can increase the bond strength without acid etching.

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Fig. 1.

Tooth surfaces were cut and polished and subsequently scanned using the laser setup as shown, bonded to composite and mounted in a shear jig for the single plane shear test.

- A. Enamel
- B. Mylar (3 mm diameter hole)
- C. Dental Stone

Fig. 2.
Setup for modified single plane shear test assembly.

Fig. 3.
Reflected light images of the laser irradiated enamel surfaces and the control samples.

Fig. 4.

Chart showing mean shear bond strength $\pm$ s.d. for the five laser groups and the control samples. Bars (groups) of the same color are statistically similar ($P > 0.05$).