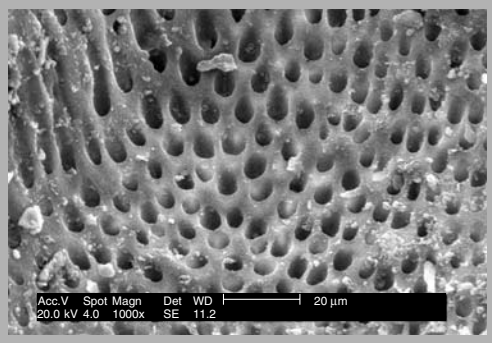
Laser Phys. Lett. **3**, No. 2 , 96–101 (2006) / **DOI** 10.1002/lapl.200510058

Abstract: Background and objective: The objective of this study was to evaluate the bond strength between a composite resin and dental hard tissues, which have been previously irradiated with an Er:YAG (2.94 μm) or CO_2 (9.6 μm) laser. Materials and methods: A total of 156 bovine teeth were divided into 6 groups: Group 1 – Enamel control: acid etched enamel (Single Bond); Group 2 – Dentin control: acid etched dentin (Single Bond); Group 3 – Irradiated enamel (CO_2 laser – 3 W) followed by acid etching; Group 4 – Irradiated dentin (CO_2 laser – 3 W) followed by acid etching; Group 5 – Irradiated enamel (Er:YAG laser – 0.16 W) followed by acid etching; Group 6 – Irradiated dentin (Er:YAG laser – 0.16 W) followed by acid etching. Results: Treatment only with acid etching and also the Er:YAG laser irradiation followed by acid etching showed the highest bond strength value while the CO_2 laser irradiation followed by acid etching treatment produces the lowest bond strength values in both tissues. Conclusion: Acid etching of non-irradiated and Erbium YAG laser irradiated tissues produces better adhesion than acid etching of CO_2 laser irradiated surfaces.



Dentin surface after CO_2 laser irradiation (150 mJ, 20 Hz, 212.2 J/cm^2, 3 W) followed by acid etching, the smooth melting pattern is removed and the dentin tubules are exposed with a similar aspect as the acid etching only

Enamel and dentin irradiation with 9.6 μm CO_2 and 2.94 μm Er:YAG lasers: bond strength evaluation

T.M. Marraccini, [1] *L. Bachmann,* [2,*] *H.A. Wigdor,* [3] *J.T. Walsh Jr.,* [4] *M.L. Turbino,* [5] *A. Stabholtz,* [6] *and D.M. Zezell* [1]

[1] Centro de Lasers e Aplicações, Instituto de Pesquisas Energéticas e Nucleares, 2242, Av. Lineu Prestes, São Paulo 05508-900, Brazil
[2] Departamento de Física e Matemática, Faculdade de Filosofia, Ciências e Letras de Ribeirão Preto, Universidade de São Paulo, 3900, Av. dos Bandeirantes, Ribeirão Preto, São Paulo 14040-901, Brazil
[3] Department of Dentistry, Advocate, Illinois Masonic Medical Center, 811, W. Wellington Ave., Chicago, Illinois 60657, USA
[4] Biomedical Engineering Department, Northwestern University, 2145, Sheridan Road Evanston, IL 60208-3107, USA
[5] Departamento de Dentística – Faculdade de Odontologia da Universidade de São Paulo, Av. Lineu Prestes, 2227 Cidade Universitária São Paulo, SP 05508-900, Brazil
[6] Department of Endodontics, The Hebrew University, Hadassah, School of Dental Medicine, P.O.Box 12272, Jerusalem 91120, Israel

Received: 13 September 2005, Accepted: 17 September 2005
Published online: 4 October 2005

Key words: adhesion; carbon dioxide laser; erbium laser; morphology; SEM; tensile strength; composite resin

PACS: 87.64.Ee, 87.19.Rr, 87.68.+z

1. Introduction

Morphological patterns produced by different treatments, such as the Erbium YAG laser, CO_2 laser or acid etching will determine the adhesion between dental hard tissues and the restorative materials. The well-established classical micro mechanical method of bonding resins consists of etching the tissue with phosphoric acid this produces an etched surface that has increased surface area to allow the resin penetration into the tooth micro-porosity. Interlocking is achieved by the penetration of the resin fluid into the tissue by capillarity action [1].

Due to the great applicability of the Er:YAG laser (2.94 μm) [2,3] and the potential application of the CO_2

* Corresponding author: e-mail: bachmann@ffclrp.usp.br

laser (9.6 μm) [4], several studies with different laser parameters have been developed to evaluate the bond strength of hard dental tissues to composite resins after irradiation with the Er:YAG laser [5–8] and the CO_2 laser [9,10]. Due to the divergent results in these studies bond strength evaluation for different laser parameters must be studied to improve the understanding of adhesion between dental hard tissue and composite resins.

One of the variables that can influence the adhesion between tissue and composite resin is the temperature rise produced by a laser on these tissues during laser irradiation. This temperature rise can change both organic and mineral matrix; and as a consequence the interlocking of tissue and restorative material [1] can no longer be efficient. Since either the surface chemical composition or microstructure in both enamel and dentin can be changed after CO_2 and Er:YAG laser irradiation, the adhesion between tissue and composite resin must be analyzed.

Laser cavity preparation can be accomplished with the Er:YAG laser or Er:YSGG laser because their wavelengths are resonant with the water molecule and hydroxyl groups [11,12]. Another laser that can be applied for cavity preparation is the CO_2 laser, its wavelength is resonant with the phosphate radical within hydroxyapatite which is the major component of enamel and dentin [11,12]. An additional laser system that can applied for cavity preparation is holmium laser [13,14]; its wavelength is resonant with water molecule, also described for the Er:YAG laser; before clinical applications of this wavelength additional experiments must be conducted.

With the increase of laser applications in dentistry the adhesive protocol performed for the conventional cavity preparation may not be efficient when the procedure of cavity preparation is performed with an Er:YAG or CO_2 laser. The aim of this study was to evaluate the bond strength between a composite resin and enamel and dentin, which have previously been irradiated with an Er:YAG laser or CO_2 laser. Visual morphological analysis will be evaluated in order to compare the effect on the surfaces of dentin and enamel after treatment with either the Er:YAG or CO_2 laser.

2. Materials and methods

2.1. Sample preparation

A total of 156 bovine maxillary incisor teeth were chosen; all teeth were free of hypoplastic areas, cracks, irregularities in enamel morphology or other dental pathology. These teeth were extracted immediately after the animals were killed, the soft tissue was removed manually and to remove the final debris the teeth were immersed in ultrasonic bath with sodium chloride solution at 0.9 wt% for 20 minutes. The crowns were separated from the roots with a diamond blade system. The crowns were individually embedded in acrylic resin and, after total polymerization, specimens were ground mechanically under water using #120 grit silicon carbide paper to remove the overlying resin and access the enamel or dentin. Finally the samples were polished with #400 and #600 grit silicon carbide paper to standardize the surface.

2.2. Sample storage

The samples were stored in sodium chloride solution at 0.9wt% for no longer then one month after extraction and until sample preparation. After the composite resin was applied, the samples were stored in distilled water at 37°C for one week and then used for the bond strength experiment. The final samples were randomly divided into six groups:

- Group 1 – Enamel control: acid etching of non-irradiated enamel;
- Group 2 – Dentin control: acid etching of non-irradiated dentin;
- Group 3 – Enamel previously irradiated with CO_2 (150 mJ, 20 Hz, 212.2 J/cm^2, 3 W) followed by acid etching;
- Group 4 – Dentin previously irradiated with CO_2 (150 mJ, 20 Hz, 212.2 J/cm^2, 3 W) followed by acid etching;
- Group 5 – Enamel previously irradiated with Er:YAG laser (80 mJ, 2 Hz, 25.7 J/cm^2, 0.16 W) followed by acid etching;
- Group 6 – Dentin previously irradiated with Er:YAG laser (80 mJ, 2 Hz, 25.7 J/cm^2, 0.16 W) followed by acid etching.

The bond strength was not evaluated in enamel and dentin that received only laser irradiation because our main objective was to evaluate the adhesion between composite resin and tissue that undergo cavity preparation with CO_2 and Er:YAG laser systems.

For the enamel samples the buccal surfaces were used and for the dentin samples the enamel was sanded until the dentin surface was evident. Twenty-six samples were prepared for each group, twenty-five samples for the tensile strength test and one sample for SEM morphological analysis.

2.3. Laser irradiation

For irradiation an Er:YAG laser (Kavo Key II, Kavo Co., Biberach, Germany) with emission wavelength at 2.94 μm and a CO_2 laser (Opus96, Opus Dent, Tel Aviv, Israel) with emission at 9.6 μm was used. In Table 1 the beam characteristics and irradiation parameters for the two laser systems are summarized. The laser treatment area had a approximate diameter of 3.5 mm.

Laser	Wave-length	Pulse width	Energy per pulse	Repetition rate	Exposure time	Focal area	Fluence	Average power
CO_2	9.6 μm	60 μs	150 mJ	20 Hz	4 s	0.3 mm	212.2 J/cm^2	3 W
Er:YAG	2.94 μm	200-500 μs	80 mJ	2 Hz	60 s	0.63 mm	25.7 J/cm^2	0.16 W

Table 1 Laser beam characteristics and irradiation parameter for the erbium and CO_2 laser systems

Procedure	Groups	Tissue	Rupture (Mpa)		Groups (p value)					
			Mean	S.D.	1	2	3	4	5	6
Acid etching	1	Enamel	15.14	0.89			3×10^{-6}		0.057	
only	2	Dentin	12.84	0.98				2×10^{-4}		0.058
CO_2	3	Enamel	9.00	0.70	3×10^{-6}				2×10^{-4}	
+ acid etching	4	Dentin	8.39	0.46		2×10^{-4}				0.011
Erbium	5	Enamel	12.93	0.70	0.057		2×10^{-4}			
+ acid etching	6	Dentin	10.53	0.67		0.058		0.011		

Table 2 Average values of rupture tensile strength with its respective standard deviations (S.D.); statistical results obtained from ANOVA analysis, evaluating the significance level (p values) of the different treatments, only the ANOVA analysis between Groups 1 and 5; and between Groups 2 and 6 does not show p value below 0.05

2.4. Acid etching

The acid etching was conducted using a kit of composite resin Single Bond Adhesive System (Z 100, 3M Dental Products Division, St. Paul, USA). This kit is composed of 3M ScotchBond 35% phosphoric acid gel, "single bond" adhesive system and Z-100 composite resin, the etching time was 15 seconds.

2.5. Composite resin

The following steps were used to form the resin cone: The samples were cleaned in an ultrasound bath for 20 minutes; 35% phosphoric acid was applied for 15 seconds, then washed with water jet for 15 seconds and dried with air jet for 10 seconds. The first layer of Single Bond was applied for 30 seconds; slight air jet was applied on the first layer, application of the second layer and photo-polymerization for 30 seconds. Immediately after the application of the single bond the resin was applied with three increments following with photo-polymerization for 40 seconds for each increment. The composite resin was inserted into a cone shape mold with the following dimensions: the small section of the resin cone was 3 mm of diameter, the larger section 4 mm and the height 4 mm. A curing light XL1500 - 3M with intensity of 400-450 mW/cm^2 was used.

2.6. Bond strength

The adhesion between the tissue and composite resin was evaluated using a tensile strength system (Instron Model 4442, Instron Corp., Canton, USA). The sample with the resin cone was fixed at the system with a loading rate of 0.5 mm/min, at rupture the load force in MPa was registered. The average value and the standard error for each group were calculated using the ANOVA analysis at 95% ($p<0.05$) significance level.

2.7. Morphological analysis

For the sample morphological analysis a scanning electron microscope (XL30, Philips, Eindhover, Holland) was used. Samples were dehydrated in alcohol solutions of increasing concentrations (50, 75, 90, and 100%) for 30 minutes for each concentration and after dehydration was achieved the samples were sputtered with gold. All dehydrated gold covered samples where kept in a dry atmosphere until the morphological evaluation.

3. Results

3.1. Bond strength

The average values of the maximum loaded tensile strength and its respective standard deviation (S.D.) are listed in the Table 2 for all groups.

The average values for the enamel and dentin can be better visualized in the Fig. 1. The "p" values obtained from the statistical analysis (ANOVA) are listed for the different treatment in the Table 2.

Using a significance level of 95% ($p<0.05$) there is statistical difference between the following groups:

- Acid etching enamel (Group 1) and enamel treated with CO_2 laser followed by acid etching (Group 3);

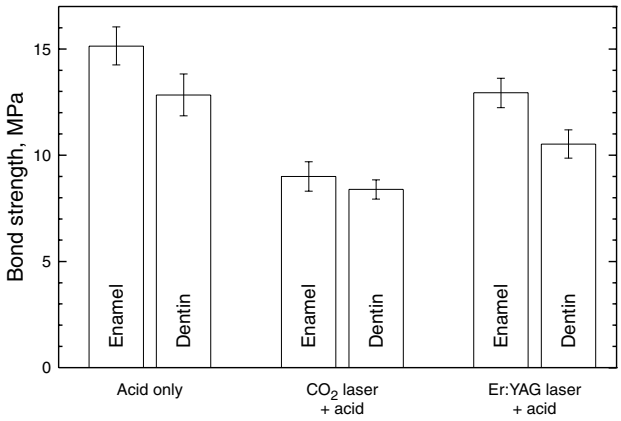


Figure 1 Average values and standard deviation of the rupture tensile strength of the enamel-resin samples: after acid etching (Group 1), CO_2 laser irradiation (150 mJ, 20 Hz, 212.2 J/cm^2, 3 W) followed by the acid etching (Group 3) and erbium laser (80 mJ, 2 Hz, 25.7 J/cm^2, 0.16 W) followed by acid etching (Group 5); and average values for dentin-resin samples: after acid etching (Group 2), CO_2 laser irradiation (150 mJ, 20 Hz, 212.2 J/cm^2, 3 W) followed by the acid etching (Group 4) and erbium laser followed by acid etching (Group 6)

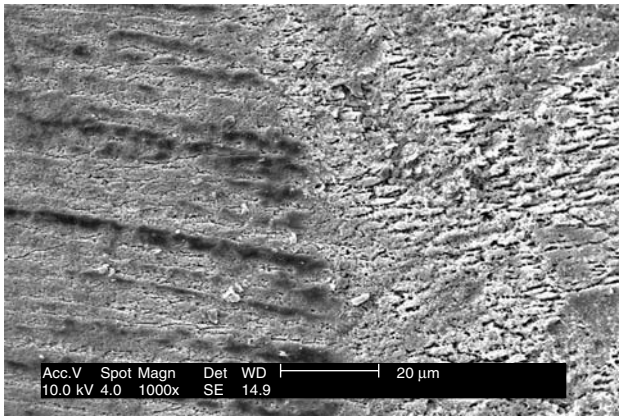


Figure 2 Enamel surface after acid etching, showing in the right side the interprismatic demineralization and in the left side the natural non-etched surface

- Enamel treated with CO_2 laser followed by acid etching (Group 3) and enamel treated with erbium laser followed by acid etching (Group 5);
- Acid etching dentin (Group 2) and dentin treated with CO_2 laser followed by acid etching (Group 4);
- Dentin treated with CO_2 laser followed by acid etching (Group 4) and dentin treated with erbium laser followed by acid etching (Group 6).

The acid etching only produces the largest bond strength for both enamel and dentin. For both tissues the

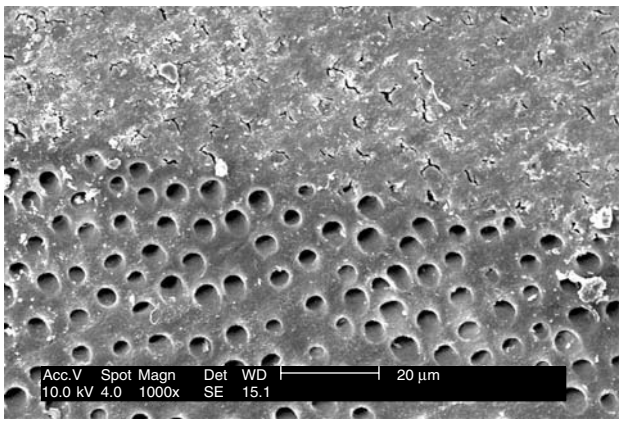


Figure 3 Dentin surface after acid etching with a dentinal tubules are exposed (bottom) and the natural non-etched surface (top)

erbium irradiation followed by acid etching and acid etching are statistically not different.

Despite the non-meaning of the bond strength comparison between the enamel and the dentin, it is possible to observe in Fig. 1 the similar behavior of the three treatments for the enamel and dentin. All three different treatments produced higher bond strength in the enamel than in the dentin.

3.2. Morphological analysis

The surface after the three etching procedures can be evaluated in the following figures. In Fig. 2 and Fig. 3 it can be observed the morphological pattern of enamel and dentin respectively after acid etching. The acid etched surfaces observed in the mentioned figures are the classical pattern that promotes an increase of resin-tissue adhesion.

The morphology of the surface irradiated with CO_2 laser followed by acid etching can be observed in the Fig. 4 for the enamel and in the Fig. 5 for the dentin. After the acid etching the prism structure is observed in the enamel surface and the dentinal tubules are exposed in the dentin surface.

For the Er:YAG irradiation followed by acid etching, the morphological pattern can be seen in Fig. 6 for the enamel and in Fig. 7 for the dentin. The morphology after the three treatments shows the prism structure in the enamel and the dental tubules in the dentin.

3.3. Discussion

The chemical composition of the tissue produces different absorption bands in the infrared absorption spectra [11], the two higher absorption regions are associated with the

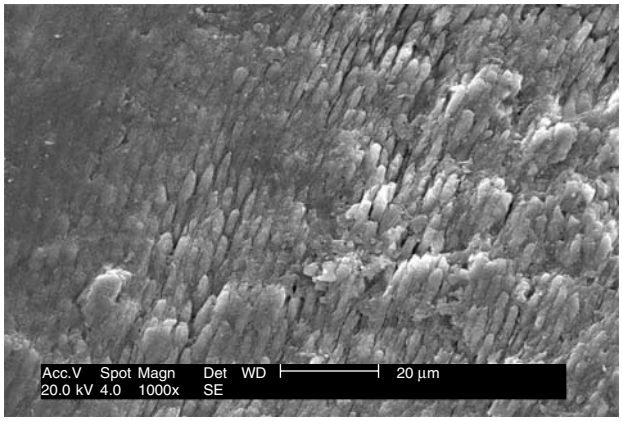


Figure 4 Enamel surface after CO_2 laser irradiation (150 mJ, 20 Hz, 212.2 J/cm^2, 3 W) followed by acid etching; the first tissue layer (melting pattern) is removed and a rough pattern is produced with a similar aspect as observed after acid etching only

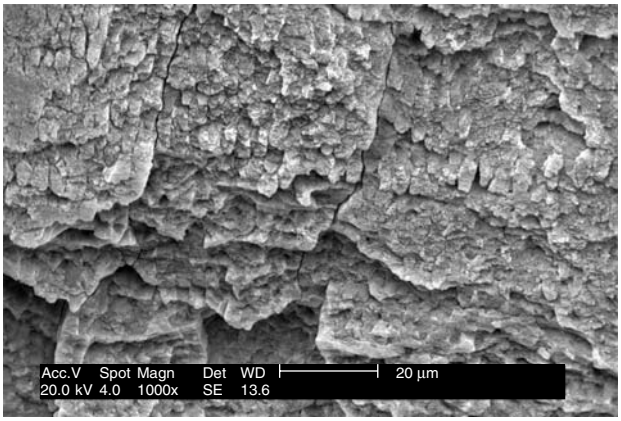


Figure 6 Enamel surface after erbium laser irradiation (80 mJ, 2 Hz, 25.7 J/cm^2, 0.16 W) followed by acid etching, the tissue layer is probably removed but a similar acid etched pattern is produced

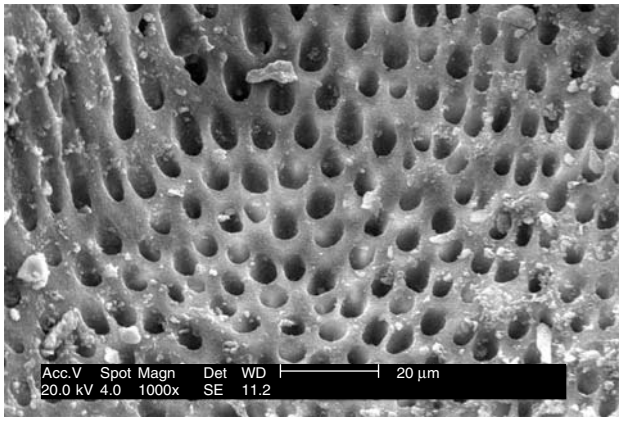


Figure 5 Dentin surface after CO_2 laser irradiation (150 mJ, 20 Hz, 212.2 J/cm^2, 3 W) followed by acid etching, the smooth melting pattern is removed and the dentin tubules are exposed with a similar aspect as the acid etching only

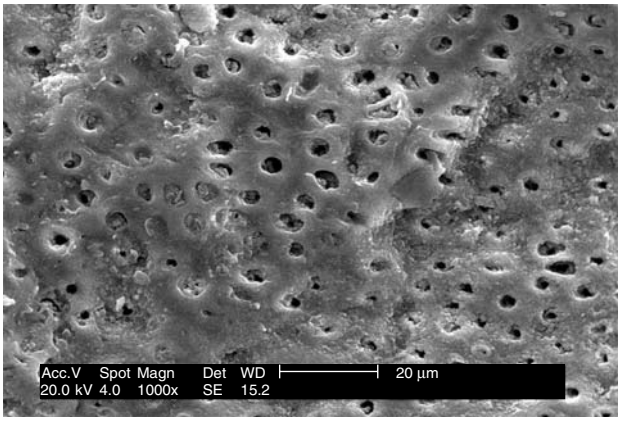


Figure 7 Dentin surface after erbium laser irradiation (80 mJ, 2 Hz, 25.7 J/cm^2, 0.16 W) followed by acid etching, the dentinal tubules are better exposed than after the erbium laser irradiation

water molecule (2-3 μm) and to the phosphate radical (9-11 μm). The closest laser wavelengths to these absorption bands are the Er:YAG laser and the CO_2 laser. The CO_2 laser irradiation of enamel with 6 J/cm^2, 100-μs-long pulses generates a temperature rise on the irradiated spot of $\sim$1000°C; an irradiation with 10 J/cm^2 results in a temperature rise above 1200°C. Er:YAG laser irradiation of enamel with 7 J/cm^2, 150-μs-long pulses produces a temperature rise of $\sim$300°C and at 9 J/cm^2 yields a surface temperature rise of $\sim$1000°C [17].

As mentioned in the section of materials and methods this research does not evaluate the bond strength without acid etching. In the literature it is possible to observe that surfaces that are laser etched only with the CO_2 or Er:YAG lasers have poorer bond strengths [8,10]. In these studies the laser etching produces lower bond strength then those observed with acid etching alone. The melted surface produced in enamel and dentin after the CO_2 irradiation is totally removed after the acid etching in both tissues. The morphology of laser irradiation followed by acid etching produces a surface similar to that seen when acid alone is used.

The morphological pattern has a great influence in the bond strength results. The composite tag formation in the tissues form the micro-capillarity tags of adhesive into the dentinal tubules and demineralized interprismatic regions. It is an important mechanism that produces an interlock between the tissues and the adhesive system. It is possible to observe that the interprismatic demineralization in enamel and the exposing of patent dentinal tubules appear in all studied groups. Therefore, the evaluation of these

two variables alone does not explain the different bond strength values. Another factor that determines good bond strength would be the demineralized inter-prismatic depth in enamel and how patent are the dentinal tubules in dentin. A quantitative evaluation is not possible with SEM microscopy alone, which allows only a qualitative morphologic evaluation.

In the dentin the presence of the organic matrix produces an interlocking with the adhesive system. In this work the presence of the organic material in the superficial regions decreases after the irradiation with erbium and CO_2 lasers [15,16], since both of them generate high temperatures on the irradiated surfaces [17]. During the irradiation there is a collagen denaturation in the superficial and sub-superficial layers of the dentin, and considering that the collagen plays an important role on the bond strength the adhesion between the dentin and composite resin is compromised.

For the dentin groups, the acid etching of the irradiated surfaces removes a superficial tissue layer and thus produces patent dentinal tubules on the erbium and CO_2 irradiated surfaces. The acid etching with 35% phosphoric acid removes approximately a dentin layer with thickness greater than 7.5 μm [18] and a enamel layer between 5 and 7 μm [19]. Despite the removal, the remaining material was still affected by the laser thermal action and thus can yet interfere in the tissue-resin adhesion. As mentioned previously the organic material is probably denatured in sub-surfaces and can compromise the bond strength.

The lower bond strength values observed here are obtained after the irradiation with the CO_2 laser that produces a higher temperature rise. Despite the fact that the superficial layer of CO_2 irradiated tissue has been removed by the phosphoric acid, a remaining sub-superficial layer was still subjected to high temperatures. The lack of statistical difference between the groups treated with acid alone and erbium laser followed by acid etching can be associated with the low temperature rise of the erbium laser irradiation. As a consequence of the low temperature rise, a low sub-superficial organic matrix denaturation occurs during irradiation and a very similar morphology pattern to the acid etching exclusively is observed [20]. For future studies obtaining better bond strength values between the lased surfaces and composite resin is necessary to evaluate different irradiation parameters, acid concentration and etching time.

The morphological pattern obtained after Er:YAG and CO_2 laser irradiation were altered after the acid etching procedure, resulting in a surface that is similar to the tissues treated with acid etching only. The bond strength values between the composite resin and the acid etched surfaces are not statistically different than the values of the erbium laser followed by acid etching treated surfaces, while the CO_2 laser followed by acid etched surfaces created the lowest bond strength values. The mean bond strength values obtained suggest that the thermal action (surface temperature rise) produced by the laser systems influence the adhesion of the tissue to the composite resin.

Acknowledgements The authors acknowledge the Centro de Lasers e Aplicações (CLA) – Instituto de Pesquisas Energéticas e Nucleares (IPEN); Faculdade de Odontologia da Universidade de São Paulo (FOUSP) – Universidade de São Paulo (USP), Northwestern University and Hebrew University for technical support and facilities. This work has a financial support from Fundação de Amparo a Pesquisa do Estado de São Paulo (FAPESP) and Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq).

References

[1] W.J. O'Brien, Dental Materials, 5th ed. (Quintessence, Chicago, 1989), pp. 71–86.
[2] R. Hibst and U. Keller, Lasers Surg. Med. **9**, 338–344 (1989).
[3] R. Hibst and U. Keller, Lasers Surg. Med. **9**, 345–351 (1989).
[4] R. Müllejans, G. Eyrich, W.H.M. Raab, and M. Frentzen, Lasers Surg. Med. **30**, 331–336 (2002).
[5] R.P. Ramos, D.T. Chimello, M.A. Chinelatti, et al., Lasers Surg. Med. **31**, 164–170 (2002).
[6] A. Martinez-Isua, L. Da Silva Dominguez, F.G. Rivera, and U.A. Santana-Penin, J. Prosthet. Dent. **84**, 280–288 (2000).
[7] L.H. Burnett, E.N. Conceição, J.E. Pelinos, and C.D. Eduardo, J. Clin. Laser Med. Surg. **19**, 199–202 (2001).
[8] J. De Munck, B. Van Meerbeek, R. Yudhira, et al., Eur. J. Oral Sci. **110**, 322–329 (2002).
[9] L.J. Walsh, D. Abood, and P.J. Brockhurst, Dent. Mater. **10**, 162–166 (1994).
[10] J.L. Drummond, H.A. Wigdor, J.T. Walsh, et al., Lasers Surg. Med. **27**, 111–118 (2000).
[11] L. Bachmann, R. Diebolder, R. Hibst, and D.M. Zezell, Appl. Spectrosc. Rev. **38**, 1–14 (2003).
[12] D. Fried, M. Zuerlein, J.D.B. Featherstone, et al., Appl. Surf. Sci. **127–129**, 852–856 (1998).
[13] D.M. Zezell, S.C. Cecchini, C.P. Eduardo, et al., J. Clin. Laser Med. Surg. **13**, 283–289 (1995).
[14] B.H. Stevens, H.O. Trowbridge, G. Harrison, and S.F. Silverton, J. Endod. **20**, 246–249 (1994).
[15] S. Kuroda and B.O. Fowler, Calcif. Tissue Int. **36**, 361–369 (1984).
[16] B.O. Fowler and S. Kuroda, Calcif. Tissue Int. **38**, 197–208 (1986).
[17] W. Seka, J.D. Featherstone, D. Fried, et al., Proc. SPIE **2672**, 144–158 (1996).
[18] M. Chiba, K. Itoh, and S. Wakumoto, Dent. Mater. J. **8**, 76–85 (1989).
[19] D.H. Pashley, Oper. Dent. **17**, 229–242 (1992).
[20] T.M. Marraccini, L. Bachmann, H.A. Wigdor, et al., Laser Phys. Lett. **2**, 551–555 (2005).