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Bond Strength of Self-Etching Primer to Bur Cut, Er,Cr:YSGG, and Er:YAG Lased Dental Surfaces

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ABSTRACT

Objective*: The purpose of this study was to evaluate the tensile bond strength of a self-etching primer system to enamel and dentin surfaces treated with Er:YAG and Er,Cr:YSGG lasers. *Background Data*: The recently introduced self-etching primer systems have been shown to adhere better to dental surfaces with thin or no smear layers. Moreover, there have been no previous reports on the bond strength of these adhesives to Er,Cr:YSGG laser-irradiated enamel and dentin, which have been shown to be free of a smear layer. *Methods*: Thirty samples of enamel and thirty of dentin were divided into three groups. The first group of each substrate served as a control with a standardized bur cut, and the other two groups were conditioned with Er:YAG (350 mJ, 10 Hz, 20 J/cm^2 for enamel; 300 mJ, 6 Hz, 17 J/cm^2 for dentin) and Er,Cr:YSGG laser (125 mJ, 20 Hz, 16 J/cm^2 for both substrates). After the bonding procedure, samples were restored with composite resin, and the tensile bond strength test was performed. *Results*: The ANOVA two-way analysis and the Tukey test at 5% significance level showed that for enamel and dentin, the bond strength values were statistically higher in Er:YAG-laser treated than in Er,Cr:YSGG-laser treated surfaces ($p = 0.0001$). However, bond strength means for both laser-irradiated groups were statistically lower than for the bur cut group (Er:YAG: $p = 0.0281$ and Er,Cr:YSGG: $p < 0.0001$). SEM observation of laser-irradiated surfaces revealed a roughened aspect and absence of smear layer. *Conclusions*: The self-etching system adhesion was influenced by the type of erbium laser used, and the bond strength was higher in the Er:YAG-laser irradiated than in the Er,Cr:YSGG-laser irradiated surfaces.

INTRODUCTION

The use of laser technology for cavity preparation and enamel and dentin conditioning has demonstrated successful results.[1–7] Among the various types of lasers currently available, erbium lasers present the best performance in this area, because their wavelengths (Er:YAG, $\lambda = 2.94$ (m; Er,Cr:YSGG, $\lambda = 2.78$ μm) are close to the peak of water absorption (Er:YAG) and hydroxyl groups (Er,Cr:YSGG).[8–10] Laser irradiation produces enamel and dentin surfaces that are free of smear layer, imbricate pattern, and crack formation. Open dentinal tubules and more prominent peritubular dentin than intertubular dentin can also be seen.[11]

In the adhesive dentistry field, many efforts have been directed towards simplifying adhesive procedures. To this end, self-etching primer systems, which do not need previous smear layer removal, were introduced. The self-etching primer systems simultaneously promote demineralization and resin infiltration through the demineralized dental surfaces. Both smear layer dilution and incorporation into the resulting hybrid layer can take place, depending on the way primer is applied.[12] The great advantage of the self-etching primers is that they are less

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technique-sensitive than the previously recommended wet-bonding technique with total etch adhesives.[13] Moreover, self-etching primers do not need to be rinsed off the tooth, which simplifies the procedure and saves clinical time.

Nevertheless, the primer contains an acidic resin monomer (pH 1.9)[14] that is less acidic than 35% phosphoric acid (pH 1.3), which results in initial demineralization that is limited in depth and extent.[13,15,16] This limited demineralization during the resin infiltration process results in thin hybrid layers of only 0.5 μm in dentin and 100 nm in enamel.[14,17]

Recently, manufacturers have attempted to increase the etching ability of self-etching primer systems, such as Clearfill SE Bond (Kuraray, Osaka, Japan), by increasing the concentration of acidic resin monomers. In spite of this increased concentration, there is still the danger that acid monomers may be buffered by the mineral contents of thick smear layers.[18] Recent reports[19,20] suggest that even some of the more aggressive versions of self-etching primers fail to etch through clinically significant thick smear layers, and unground aprismatic enamel, resulting in decreased tensile bond strengths.[13,21] These findings suggest that self-etching primers should be used *in vivo* with a surface preparation method that creates thin or even no smear layers. Therefore, the hypothesis to be tested was whether dental surfaces produced by Er:YAG and Er,Cr:YSGG laser irradiation, which have been shown to have no smear layer and good permeability, could produce more effective bonding of the self-etching primer systems. As this was the first bond strength study utilizing Er,Cr:YSGG laser irradiation with self-etching primers, it was decided to start by testing the manufacturer's suggested settings.

MATERIALS AND METHODS

The present study was approved by the University of São Paulo Ethics Committee (Process number 86/04). Seventy-two freshly extracted human third molars with no caries or fillings were selected and kept in distilled water. The roots were separated and the crowns were cut in the mesio-distal direction using a diamond saw under refrigeration (Fig. 1a). The teeth were randomly divided into six groups, three for enamel and three for dentin:

Enamel specimen preparation

Lingual or buccal enamel surfaces were flattened and then embedded in acrylic resin. Using an automatic polishing machine, the specimens were polished flat using 240 and 400 grit for surface exposure, and finally 600 grit for polishing with silicon carbide waterproof abrasive paper under running water for 60 seconds. A large amount of water was used to prevent acrylic resin from becoming embedded into the enamel surface during the grinding procedure. These 30 enamel samples were divided in three groups (n = 10) and grouped as follows:

Group EC: standard bur cut with cylinder diamond burs with medium-sized particles
Group EYAG: conditioning with Er:YAG laser (description in Table 1)
Group EYSGG: conditioning with Er,Cr:YSGG laser (description in Table 1)

Dentin specimen preparation

The buccal enamel of the molars was removed using a slow speed diamond saw (Fig. 1b). Dentin surfaces were inspected for absence of enamel using a stereomicroscope. The teeth were embedded in acrylic resin and then polished as previously described for enamel. These 30 dentin samples were divided into three groups (n = 10) in and grouped as follows:

Group DC: standard bur cut with cylinder diamond burs with medium-sized particles
Group DYAG: conditioning with Er:YAG laser (description in Table 1)
Group DYSGG: conditioning with Er,Cr:YSGG laser (description in Table 1)

All the settings used in this study were selected in accordance with each laser manufacturer's instructions. The irradiations were made with the beam aligned perpendicular to the surface and moved in a sweeping fashion by hand during the exposure period. In order to fix the working distance, a k-file was adapted to the hand piece head (Fig. 1c) The Er:YAG laser used was the Kavo Key Laser 3 (Kavo Dental GmbH & Co. AU1 KG,) and the Er,Cr:YSGG laser used was the Millennium (Biolase Technology Inc.,). A complete description of the laser parameters is summarized in Table 1.

All teeth were subjected to the same bonding procedure using the Clearfill SE Bond system (Kuraray, Osaka, Japan) following the manufacturer's instructions exactly. Primer was applied for 20 seconds, gently air dried, and then the bond was applied and photopolymerized for 10 seconds. The composition and classification[17] of the adhesive used in this study is listed in Table 2. The bonding area was 7.07 mm.[2]

The embedded specimens were fixed in a metal clamping device (developed by Houston Biomaterials Research). A split bisected Teflon matrix was placed over the tooth/resin block resulting in an inverted conical cavity of 3 mm diameter at the bottom, 5.6 mm at the top, and 3 mm deep (Fig. 1d).

The hybrid photopolymerized composite resin (Filtek Z-250; 3M Dental Products, St. Paul, MN, USA) was inserted in the matrix in three increments and each was polymerized for 20 seconds. When the matrix cavity was completely filled, the specimen was removed from the clamping device and the matrix was opened and separated, leaving an inverted composite resin cone adhered to the enamel surface.

After 24 hours of storage in distilled water at 37°C, the samples were submitted to tensile bond strength test in a universal testing machine at a speed of 0.5 mm/min until fracturing occurred (Fig. 1e) The resulting tensile bond strength values (in MPa) were subjected to statistical analysis.

This materials and methods followed the ISO Standard: Dental Materials Testing of Adhesion to Tooth Structure (ISO/TS: 11405-2003; dental materials—testing of adhesion to tooth structure).

Scanning electron microscopy (SEM) evaluation

Two extra samples of each group were laser irradiated or bur cut, gradually dehydrated, and covered with a thin layer of gold

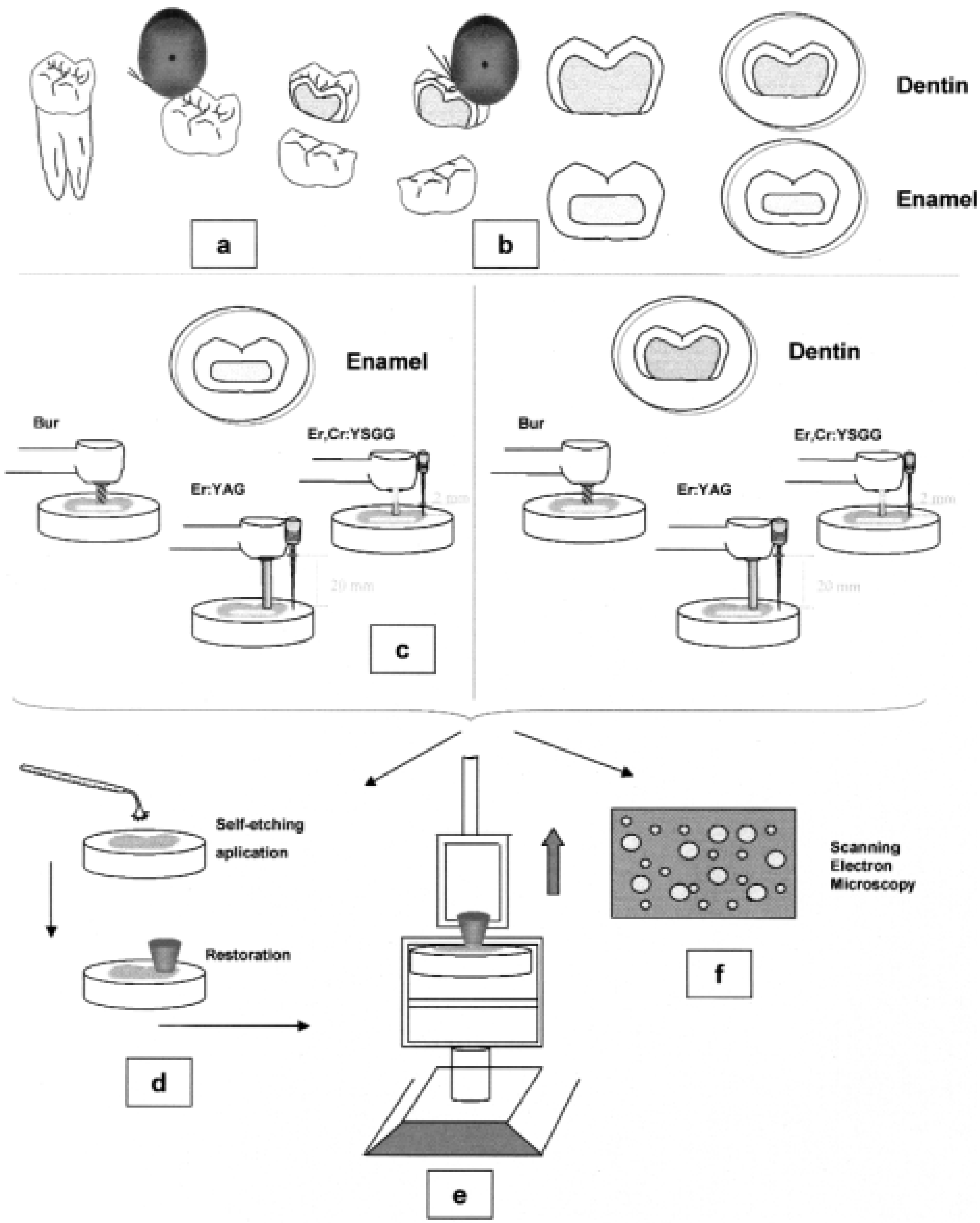

FIG. 1. (**a**) The crowns were cut in the mesio-distal direction and one of the halves obtained was used for the enamel study and the other for the dentin study. (**b**) To expose superficial dentin, the outer buccal enamel was cut with a diamond saw, and to expose flat enamel it was polished. (**c**) Embedded and polished samples were irradiated with the beam aligned perpendicular to the surface, and the working distance was fixed using a k-file adapted to the hand piece head. (**d**) Adhesive procedure and inverted conical restorations. (**e**) Tensile bond strength test after 24 hours of storage in distilled water. (**f**) Scanning electron microscopy was performed immediately after irradiation before bonding was done.

Table 1. Description of Both Lasers and the Parameters Used for Each Substrate

	Enamel		*Dentin*	
	Er:YAG	*Er, Cr:YSGG*	*Er:YAG*	*Er, Cr:YSGG*
Wavelengths	2.94 μm	2.78 μm	2.94 μm	2.78 μm
Pulse duration	250–500 μs	140–200 μs	250–500 μs	140–200 μs
Hand piece	2065	—	2065	—
Fiber	—	Z6	—	Z6
Energy per pulse	350 mJ	125 mJ	300 mJ	125 mJ
Repetition rate	10 Hz	20 Hz	6 Hz	20 Hz
Fluency	20 J/cm^2	16 J/cm^2	17 J/cm^2	16 J/cm^2
Working distance	20 mm	2 mm	20 mm	2 mm
Irradiation time	30 s	15 s	30 s	15 s
Focal spot	1.5 mm	0.49 mm	1.5 mm	0.49 mm
Water spray	0.1 mL/s	0.7 mL/s	0.1 mL/s	0.7 mL/s

to be examined in the scanning electron microscope (XL300; Phillips, Eindhoven, Netherlands) (Fig. 1e and f).

RESULTS

Tensile bond strength

The mean tensile bond strength and standard deviations are summarized per experimental group in Table 3. The mean tensile bond strength values ranged from 7.78 MPa (Er,Cr:YSGG conditioned dentin) to 20.66 MPa (bur cut enamel).

These data were submitted to analysis of variance (ANOVA two-way) at 5% significance level. The analysis showed statistically significant difference between treatments ($p < 0.0001$) between the substrates ($p < 0.0001$) and there was no significant interaction between factors ($p = 0.45755$).

The following Tukey test between substrates, at 5% significance level, showed that bond strength to enamel was significantly higher than to dentin ($p < 0.0001$). The comparison of the different treatments showed that the tensile bond strength was significantly higher in the control groups treated with bur cut than in the groups treated with laser (Er:YAG, $p = 0.0281$ and Er,Cr:YSGG, $p < 0.0001$). Moreover, the bond strength to Er:YAG-treated surfaces was significantly higher than to the Er,Cr:YSGG-treated surfaces ($p = 0.0001$).

SEM

The micrographs of bur cut surfaces showed the presence of a smear layer that occluded the enamel prisms or dentinal tubules (Fig. 2a and d). Both laser-irradiated enamel surfaces were free of a smear layer and had a rough aspect. Their appearance was similar to etching pattern type I, and had exposed prisms with deep central excavations and prominent margins, and these were more common in the Er,Cr:YSGG-lased enamel surfaces (Fig. 2b and c).

The two laser-irradiated dentin samples revealed rough surfaces with opened dentinal tubules, an absence of a smear layer, and more prominent peritubular dentin than intertubular dentin (Fig. 2e and f). The Er:YAG-irradiated surfaces, however, presented more opened tubules (Fig. 2e).

DISCUSSION

To the authors' knowledge, there are currently no comparable studies analyzing the influence of Er,Cr:YSGG laser irradiation on the bond strength of a self-etching system. Earlier studies were conducted exclusively using an Er:YAG laser, and most of them were associated with phosphoric acid. The few studies that analyzed the bond strength of self-etching systems after Er:YAG irradiation or diamond bur cutting revealed similar bonding to enamel, although in dentin the bond values of the lased surfaces were statistically lower.[11,22]

In contrast with those authors, in the present study we found that in both enamel and dentin, Er:YAG laser irradiation produced lower bond strength means than those of the bur-prepared surfaces, when they were used with Clearfill SE Bond. The same occurred with Er,Cr:YSGG laser irradiation, but for this laser the resulting bond values in both substrates were also significantly lower than those resulting from Er:YAG irradiation. The groups treated with this laser system showed the lowest means: 11.24 ± 2.9 MPa in enamel and 7.78 ± 2.61 MPa in dentin.

In contrast to the bur cut surfaces that were covered with a smear layer, both Er:YAG and Er,Cr:YSGG laser irradiated

Table 2. Composition of the Adhesive System Used

Product	*Primer*	*Adhesive*	*pH*	*Classification*
Clearfill SE Bond	10-MDP, HEMA, water	MDP, HEMA, Bis-GMA, microfiller, photoinitiator	~2	Mild self-etch adhesive

10-MDP: 10-methacryloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol glycidyl methacrylate.

TABLE 3. MEAN BOND STRENGTH VALUES AND STANDARD DEVIATION OF DIFFERENT GROUPS

Clearfill SE Bond	*But cut*	*Er:YAG*	*Er, Cr:YSGG*
Enamel	20.66 (± 2.78)[a]	17.93 (± 3.56)[b]	11.24 (± 2.90)[c]
Dentin	15.12 (± 3.95)[a]	11.56 (± 5.78)[b]	7.78 (± 2.61)[c]

Means with the same superscript letter show no statistically significant difference ($p < 0.05$).

enamel surfaces showed the absence of a smear layer, and had more roughness, a microretentive pattern, and prism exposure. In dentin, open dentinal tubules, absence of a smear layer, and more prominent peritubular dentin than intertubular dentin were observed. These surface patterns are normally seen after traditional acid conditioning and are expected to promote good bonding.

However, during laser irradiation there are other simultaneous effects that can interfere with bonding, even when the surfaces are mechanically appropriate. In the case of dentin, the known collagen thermal degradation after laser irradiation could be one of the factors involved.[23,24] Although in some short-term studies, deproteinized dentin (without collagen) has shown better bonding than protein-preserved dentin, but no long-term clinical evidence of this has been shown.[25] Moreover, in enamel, which has only a very small amount of protein (0.5 wt%),[26] the same decreased adhesion is observed.

Even if the absence of a sound collagen network did not interfere with bonding, it has been demonstrated that the denatured organic matrix blocks the diffusion pathways in enamel and dentin. The diffusion pathway blockage affects the porosity of the structures and consequently impairs penetration of the adhesive components.[27] An adhesive flow depth of several nanometers into the demineralized enamel and dentin is fundamental to promoting the necessary micromechanical retention.

Moreover, after laser irradiation the dental surfaces are chemically modified. Irradiation with erbium lasers promotes loss of carbonate, formation of new hydroxyapatite-like crystals, and consequently more acid-resistant surfaces.[23,28,29] Possibly, in the presence of this more acid-resistant surface, the weak acids present in the self-etching system cannot sufficiently modify the surface to promote adhesive penetration.

The different behavior of the two laser systems could be a consequence of the different tissue interactions. The absorption at 2.78 μm is mainly due to OH^- (hydroxyl groups from hard mineralized tissue), so that during explosive ablation, some of these hydroxyl groups can absorb the incident light, causing higher surface temperatures at the ablation threshold than are seen with the 2.94-μm Er:YAG irradiation, which is mainly absorbed by water.[30] Thus the Er,Cr:YSGG laser could have caused a higher rise in surface temperature than the Er:YAG laser, and as a result more chemical surface alteration occurred, since these alterations are extremely temperature dependent.

As this study utilized the settings suggested by the manufacturer's manual, for the Er,Cr:YSGG laser system the same settings were used for both enamel and dentin. The 125-mJ energy level (and 2 mm working distance) used seems to be too high for dentin. It is known that for any type of teeth (deciduous or permanent), the same erbium laser settings will interact more with dentin than with enamel.[31] Because of the lower water content in enamel, this substrate always requires higher energies. Therefore different laser settings are recommended for these different surfaces. Furthermore, the pulse width could be modified in order to obtain better results, since shorter Er,Cr:YSGG laser pulses (around 5 μs) minimize thermal damage, enabling better bonding to restorative materials.[32]

Further studies should be carried out to find the best enamel and dentin conditioning settings for the Er,Cr:YSGG laser, since in a previous study,[4] using the total-etch technique instead of a self-etching system, one Er,Cr:YSGG setting (150 mJ and 20 Hz) was found to provide enamel bond strength means similar to those of bur-prepared surfaces.

In the present investigation, a conventional tensile bond strength test was chosen for bonding evaluation. Both tensile bond strength and microtensile bond strength (μTBS) tests have limitations and do not allow a reliable link to be established between laboratory bond strength data and clinical performance.[33,34] The μTBS was introduced to overcome the limitations of the traditional shear and tensile bond strength tests. Due to the very small cross-sectional bond areas, a more uniform load is induced in the microtensile technique. However, skillful investigators are required and the technique is very sensitive, especially when enamel is considered. The stick cutting procedures transmit vibrations to the specimens and create microfractures that can lead to premature failure.[35,36] In particular, enamel may be intrinsically too weak (friable) to withstand the stress induced during the creation of such small sticks. Thus it is common for the μTBS values obtained in enamel to be lower than those seen in dentin.[13,36] This observation contradicts years of laboratory and clinical evidence showing that bonding to enamel is more reliable than bonding to dentin.[17,37,38] Therefore, μTBS testing of enamel specimens results in bond strength values that are strongly influenced by the technical procedure used, and may reflect the propagation of microfractures rather than the adhesive interface bond strength.

In the present study, since adhesion to enamel was also tested and compared with dentin values, it was decided to use a tensile bond strength test. This methodology is less time- and resource-consuming and could address the authors' primary objective, which was to evaluate whether it was possible to obtain bond strength values for a self-etching primer system used with erbium laser-treated surfaces that was similar to the bonding values seen in bur-treated surfaces. This objective was achieved and the comparison was possible, although the mean bond strength values to dentin were probably lower than they would be if they had been obtained with the μTBS method.

In spite of the current problems with bonding to laser-treated dental tissues, efforts should be made to enhance it, as laser treatment has the great advantage of resulting in decontaminated surfaces.[39] The presence of bacteria impairs the biological effectiveness of the restoration, and also introduces defects

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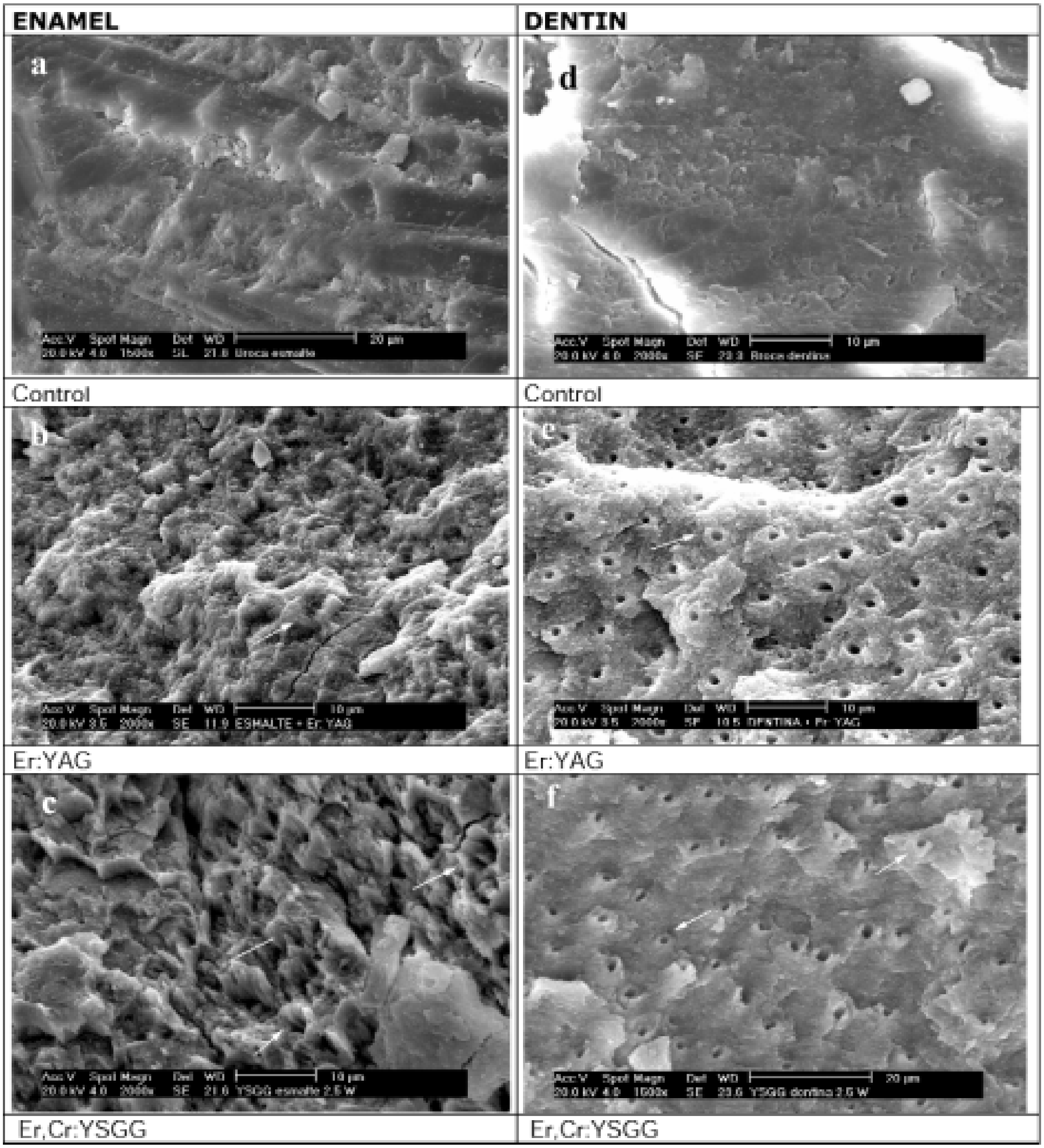

FIG. 2. (**a**) Control enamel surface showing presence of a smear layer and absence of exposed prisms. (**b**) Enamel irradiated with Er:YAG laser (20 J/cm^2, 350 mJ, 10Hz), showing a rough surface free of a smear layer and some areas with a type I etching pattern (deep central excavations and prominent margins) (arrow). (**c**) Enamel irradiated with Er,Cr:YSGG (16 J/cm^2, 125 mJ, 20 Hz), also showing a rough surface and overall type I etching pattern (arrows) (**d**) Control dentin surface showing presence of a smear layer and closed dentinal tubules. (**e**) Dentin irradiated with Er:YAG laser (17 J/cm^2, 300 mJ, 6 Hz), showing wide open dentinal tubules with more prominent peritubular dentin than intertubular dentin (arrows). (**f**) Dentin irradiated with Er,Cr:YSGG laser (16 J/cm^2, 125 mJ, 20 Hz), showing more prominent peritubular dentin than intertubular dentin (arrows) and fewer open tubules than the Er:YAG-laser irradiated dentin (original magnification 2000×).

that weaken bonding, thus compromising the overall result of the restorative treatment.[40] Therefore, this is a great advantage of lasers and should not be ignored by modern restorative dentistry.

It should be noted that although one representative self-etching adhesive system was used, only one set of irradiation parameters for each laser and each substrate were tested. Therefore, further studies testing a greater variety of settings are required. Moreover, as there are many clinicians that would buy a laser device and use it by following the instructions in its manual, it is very important for these systems to come with reliable setting suggestions.

CONCLUSION

The hypothesis tested in this study, that better bond strength of the new self-etching systems could be expected after Er:YAG and Er,Cr:YSGG laser treatment in both enamel and dentin, was not confirmed. However, the self-etching system had higher bond strengths for the Er:YAG-laser irradiated than for the Er,Cr:YSGG-laser irradiated surfaces.

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