

Build-Up of a Resin Composite Core in a Fiber-Reinforced Post by a 2.78 μm -Pulsed Laser Treatment

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This study aims to evaluate the effect of the surface treatment of fiber-reinforced posts through different treatment methods, including 2.78 μm erbium chromium: yttrium scandium gallium garnet (Er, Cr: YSGG) laser at different powers, on the push-out bond strength of methacrylate resin-based glass fiber reinforced composite post to core composite. To obtain clinical success in post-supported restoration, a durable bond between post and core interfaces is very important. The effect of the Er Cr: YSGG laser on bonding strength when applied as a surface pretreatment of post surface has not been studied sufficiently. Forty-two fiber-reinforced posts were randomly divided into six groups according to surface treatment: Group 1: control with no surface treatment; Group 2: coated with silicated alumina particles (Co-Jet system, 3M ESPE, Seefeld, Germany); Group 3: 9% hydrofluoric (HF) acid for 1 min; and Groups 4, 5, and 6: 1 W, 2 W, and 3 W Er, Cr: YSGG laser for 1 min, respectively. After the application of the silane coupling agent to the surface of the posts, core material was applied to the posts, and three slices were obtained from each sample to evaluate the bond strength using the push-out test ($n=18$). The Co-Jet sandblasting group showed the highest values, while HF acid and 1 W and 2 W Er, Cr: YSGG laser treatments enhanced the bond strength between the fiber post and the composite core ($p<0.05$). Surface treatments of fiber-reinforced posts could enhance the bond strength to attain composite core build-up. Nevertheless, lower power settings might be preferred when using the hard-tissue laser. A chairside Er,Cr:YSGG laser system commonly preferred in dental applications increase the bond strength at lower power settings.

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1. Introduction

Insufficient coronal structure of a tooth due to serious damage by decay and/or traumatic dental injuries commonly requires the placement of a post system inside the root canal following an adequate endodontic treatment to provide a retentive mean for the core and coronal restoration [1]. Practitioners commonly use prefabricated or custom posts made of metal, which increases the risk of unfavorable vertical root fracture because their elastic modulus is above that of the dentin [2]. In the 1990s, fiber-reinforced composite (FRC) posts with elastic properties similar to those of dentin (approximately 20 GPA) were introduced, and these FRC posts make fractures restorable [2, 3]. Moreover, this type of post overcomes corrosion and biocompatibility problems associated with metal posts [4]. These properties of FRC posts, which are composed of reinforced carbon, quartz, or glass fibers embedded in epoxy or a methacrylate resin polymerized matrix, make them advantageous in restoring endodontically treated teeth [5].

The achievement of reliable bonds between post and core interfaces is important to the clinical success of post-supported restoration. The retention of core materials to the post is affected by various factors, such as post shape and design, choice of post and core materials, and surface pre-

treatment methods of posts [6, 7]. Surface treatment with different conditioning techniques is a common method for improving the bond strength between posts and composite resin cores [8]. For this purpose, micro-mechanical and chemical surface treatment approaches have been proposed [9, 10]. Currently, acid etching of the post surface and coating of the post surface with silane are preferred as chemical approaches because of their ability to promote adhesion between organic and inorganic compound. The application of silane on post surfaces has also been widely investigated with contradictory results [11]. Another application, the tribochemical silica coating called the Co-Jet system, uses aluminum oxide particles modified by silica. In the Co-Jet process, the bonding area is increased, and a silicate layer is then welded onto the post surface. The silane can be applied to the tribochemical-coated surface to obtain combined chemical and micro-mechanical retention [9].

Recently, surface treatment with a 2940 nm wavelength erbium: yttrium aluminum garnet (Er: YAG) laser has been found to be effective in improving the bond strength between the FRC post and the core material [12]. However, it remains unknown if differences in absorption, wavelength, pulse duration, and ablation rate of the Er, Cr: YSGG laser (2780 nm) would improve bond strength. As regards the Er, Cr: YSGG laser treatment of FRC posts, no experimental

research has been undertaken to date. Therefore, this study aims to evaluate the effect of surface treatments with the Er, Cr: YSGG laser at different parameters and compares the effects between Co-Jet sandblasting and hydrofluoric (HF) acid on the push-out bond strength of methacrylate resin-based glass fiber-reinforced composite posts to core material. The null hypothesis states that no difference exists in the various surface treatments in bond strength between the FRC post and core material.

2. Materials and Methods

2.1 Sample Preparation

Forty-two methacrylate resin-based glass fiber-reinforced composite posts (Rebilda post, VOCO, Cuxhaven, Germany) with a diameter of 2 mm were used in the study. The FRC posts were randomly distributed into six groups as follows:

Group 1 (Control): No treatment.

Group 2: The specimens were sandblasted by silicate-coated alumina particles (Co-Jet system; 3M ESPE, St. Paul, MN, USA) with a diameter of 30 μm at a pressure of 2.3 bar (2.3 Pa \times 10⁵ Pa) and from a distance of 10 mm.

Group 3: The specimens were treated with 9% buffered HF acid (Ultradent, South Jordan, Utah, USA).

Group 4: The specimens were treated using the Er, Cr: YSGG (Waterlase iPlus, Biolase Technology Inc., CA, USA) laser with 2.78 μm wavelength, pulse duration of 140 μs to 200 μs , repetition rate of 10 Hz, and output power of 1W. A 600 μm -diameter laser optical fiber was aligned perpendicularly to the fiber post surface at a 1 mm distance and scanned the whole post area. The energy parameters at 1 W and water/air flow of 70% and 60%, respectively, were used continuously during the irradiation.

Group 5: The specimens were treated by the Er, Cr: YSGG laser with an average power of 2 W and at a repetition rate of 10 Hz.

Group 6: The specimens were treated by the Er, Cr: YSGG laser with an average power of 3 W and at a repetition rate of 10 Hz.

All of the above treatments for posts were performed for a duration of 1 minute. Then, the posts were thoroughly rinsed with water spray and dried with oil-free air. The silane coupling agent (Monobond Plus; Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied on the surface of the FRC posts with a microbrush and was left on the surface for 1 min according to the manufacturer's instructions. Afterwards, the excess was dispersed with a strong stream of air.

Following the surface treatments, the FRC posts were built up to a composite core material (Grandio Core DC, VOCO, Cuxhaven, Germany). The FRC posts are tapered at the apical part of their design. Only the parallel parts of the posts were used to simplify the calculation of the bonded area. Each FRC post was perpendicularly placed on a cylindrical Teflon mold with 10 mm diameter maintained at the coronal portion. An incremental technique was applied to build up the core. Each 2 mm increment of the core composite was cured for 40 s using a light-curing unit (Valo; Ultradent, Salt Lake City, Utah, USA) with an output of 1 W/cm² according to the manufacturer's instruction. The mold was subsequently removed, and the core material was additionally light-cured for 40 s to ensure optimum

polymerization. The main compositions of the post, silane, and composite resin core are listed in Table 1.

Table 1 – Materials used in this study

Material	Product	Composition	Manufacturer	Lot
Composite Core Build-up	Grandio Core DC	Matrix: Bis-GMA, UDMA resins. Filler: silica/Ba-glass ceramics (77% wt), amines, benzoyl peroxide, BHT	VOCO, Cuxhaven,	1310229
FRC post	Rebilda post *	70% glass fiber, 10% filler, 20% UDMA	Germany	1339313

Bis-GMA: bisphenol A-glycidyl methacrylate, UDMA: urethane dimethacrylate, Ba: barium, BHT: butylated hydroxytoluene.

*:Size # \varnothing 2

After storing the specimens in distilled water at 37 °C for 24 h, they were cut along the perpendicular long axis of the post to obtain three slices of 2 mm in thickness by a precision saw (Isomet1000, Buehler, Lake Bluff, IL, USA) at a low speed with water cooling. Finally, 18 samples were prepared for the push-out bond strength test from each group (n=18). The definitive thicknesses of the slices were recorded after measurement using a digital caliper. Moreover, the upper and lower diameters of each post were measured and recorded. In Fig. 1, a detailed illustration represents the test methods. The figure represents the laser application and push-out bond strength test methods. First, the FRC post was placed in the laser application device that allows the movement of the laser tip at a certain speed and direction. The post was placed directly below the laser tip horizontally on device, in this way, the laser irradiation was allowed to contact the entire surface of the fiber post. Afterwards, the FRC post was placed in center of the Teflon mold and the resin core was built up. Post-core slices at 2 mm in height were cut. Finally, a disk was obtained consisting fiber-post at the middle and the push-out test was performed. The images of FRC post and core samples before and after the push out test were represented.

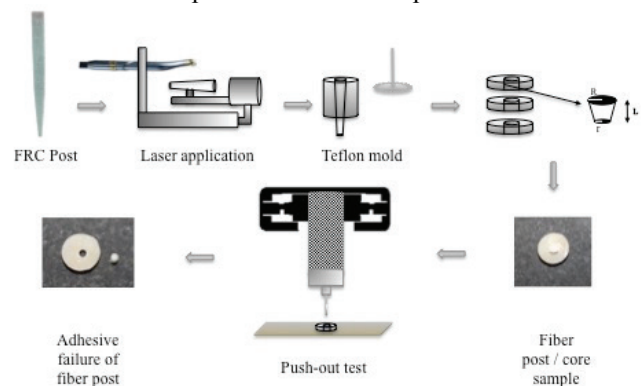


Fig. 1. Schematic representation of the test procedures.

2.2 Push-out Bond Strength Test

A push-out test was performed on each specimen with a universal test machine (AGS-X; Shimadzu Corporation; Tokyo; Japan) at a crosshead speed of 0.5 mm per minute. The maximum load at failure was recorded in N unit and was recorded in N unit and was divided across the bonded area to convert to MPa unit. The adhesion area of the FRC post was calculated using the following equation:

$$A = (R + r)\pi\sqrt{(R - r)^2 + L^2}$$

where r and R are the smallest and largest diameters of the cross-sectioned tapered post, respectively, and L is the thickness of the section.

After the test procedure, each specimen was visually examined under the stereomicroscope (Zeiss Stemi 2000C; Carl Zeiss; Jena; Germany) at 32× magnification to evaluate the failure mode. Three types of failure were classified: adhesive failure (post surface exposed without the core material), cohesive failure (fracture within the FRC post or core material), and mixed (a combination of the two: cohesive and adhesive).

2.3 Scanning Electron Microscopy (SEM) Analysis

After the samples were dried and coated with gold and palladium by sputter coating (Polaron SC7620 Sputter Coater, VG Microtech, West Sussex, England), they were observed by a scanning electron microscope (Zeiss-Leo 1430 SEM, Angstrom Scientific Inc., NJ, USA) with magnifications of 100× and 500×.

2.4 Statistical Analysis

The results were statistically analyzed by Kruskal–Wallis test, and the Mann–Whitney U test was used to conduct pairwise comparisons in the push-out bond strength data (MPa) using specific software (IBM SPSS Statistic 20.0 for Mac). A 95% confidence level was used.

3. Result

The mean push-out bond strength and standard deviations are 17.70 ± 3.53 MPa for the control group. All surface treatments showed statistically significant higher bond strength than the control group ($p < 0.05$) except the 3 W Er, Cr: YSGG laser treatment group, which has 20.37 ± 7.72 MPa. The Co-Jet sandblasting treatment group produced 28.76 ± 5.15 MPa and the highest bond strength value ($p < 0.05$). The HF acid surface conditioning group produced 23.78 ± 5.35 MPa and displayed enhanced bond strength compared with the control group ($p < 0.05$). Moreover, the 1 W, 2 W, and 3 W Er, Cr: YSGG laser treatments produced 23.19 ± 5.24 MPa, 20.93 ± 5.53 MPa, and 20.37 ± 7.72 MPa, respectively, and the laser treatment group had higher bond strength values than the control. However, no statistical significant difference was found in the 3 W laser power setting group.

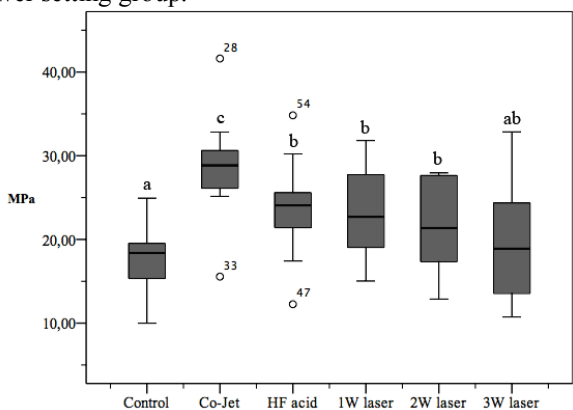


Fig. 2. Box plot of the bond strength values from the push-out test.

The statistical differences among the groups and the box plot diagram of the bond strength values are represented in Fig. 2. (Different lowercases indicate statistically different groups according to the Mann–Whitney U analyses. $p < 0.05$)

Fig. 3 shows the distribution of the failure pattern for each of the six groups. For the control group and the laser-treated groups at 2 W and 3 W, adhesive failure had the highest percentage than the other failure types. However, in the other groups, cohesive and mixed failures were predominant.

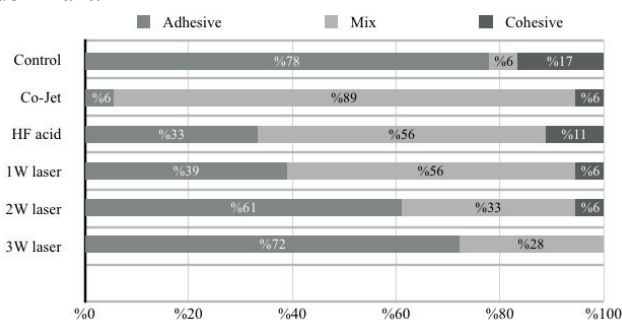


Fig. 3. Failure pattern distribution among the groups tested.

Additionally Fig. 4 represents the failure types of samples. In Fig. 4A, the bonding between the resin composite and the fiber post debonded from the adhesive joint. No residual resin composite on the fiber post surface was observed. However, as shown in Fig. 4B, surface cracks of the fiber post can be observed because of cohesive failure. Fig. 4C shows the mixed failure pattern.

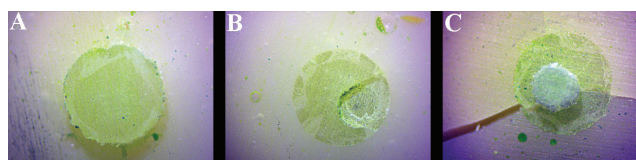


Fig. 4. Representative images of the failure types: A) adhesive between the post and core material, B) cohesive in the post, C) mixed failure.

SEM evaluation revealed no changes in the control group. The Co-Jet sandblasting group showed a rough surface topography and a micro-retentive silica coating surface was observed. HF acid showed partial and superficial removal of the methacrylate resin matrix on the post surface, and 1 W Er, Cr: YSGG laser showed better removal of the resin matrix than HF acid. The 2 W Er, Cr: YSGG laser application removed a huge amount of the resin matrix. In the 3 W Er, Cr: YSGG laser group application, the fibers were scattered and ruptured, and remains of the methacrylate resin matrix surrounding the fibers were not found. In Fig.5, SEM images of samples from each group are represented.

4. Discussion

The use of different surface treatments influenced the bond strength between the fiber posts and the core build-up materials. Silica-coated alumina particle abrasion, acid etching, and laser treatments significantly enhanced interfacial bonding. Therefore, the null hypothesis that states that no difference exists among the various post surface treatments in bond strength between the fiber and the core material is rejected.

In the present study, the bond strength between the FRC post and the core material after various treatments was evaluated using the push-out method. This method is commonly used for bond strength evaluation [13-15], and it provides a uniform stress distribution by reducing the dimension of the samples [14].

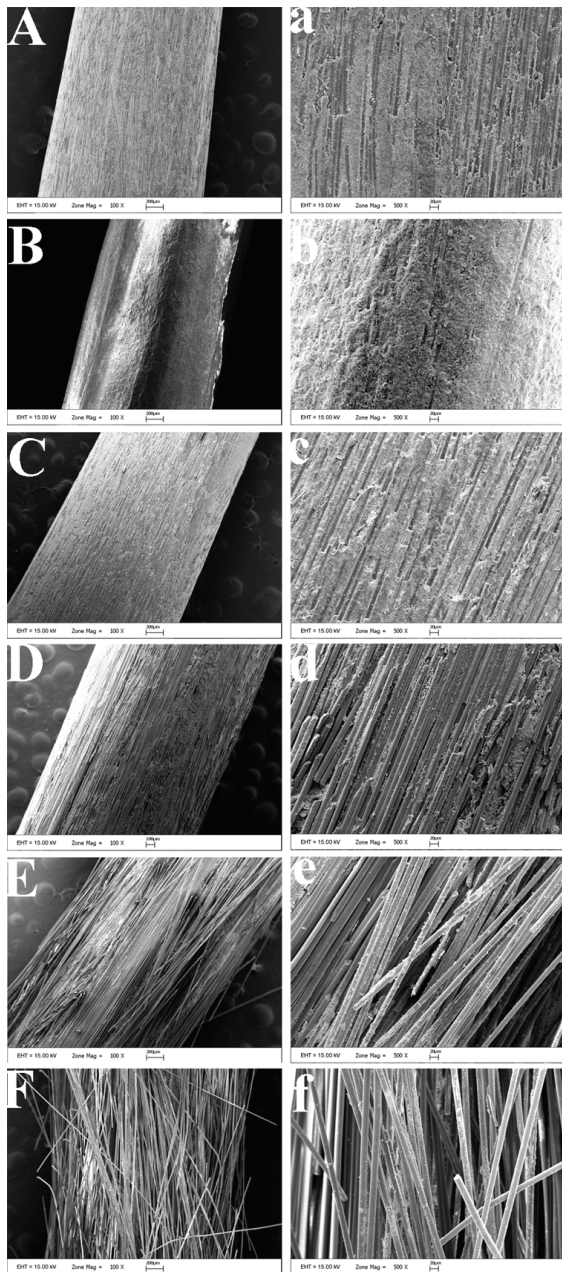


Fig. 5: SEM images of the FRC post surfaces after different treatments at a magnification of 100 \times (uppercase letter) and 500 \times (lowercase letter).

- A-a: Untreated FRC post surface; no changes
 B-b: Co-Jet sandblasting treatments; rough and micro-retentive silica coating surface topography
 C-c: HF acid; partial removal of the methacrylate resin matrix on the post surface
 D-d: 1 W Er, Cr: YSGG laser; removes more resin matrix than HF acid
 E-e: 2 W Er, Cr: YSGG laser; removes a huge amount of resin matrix
 F-f: 3 W Er, Cr: YSGG laser; the fibers were scattered and ruptured, no remains of the methacrylate resin matrix surrounding the fibers were observed

As bonding effectiveness between the fiber-reinforced post and the composite core is crucial for the longevity of endodontically restored teeth, many studies have focused on the surface treatment of the fiber-reinforced post to improve bonding strength. The most common micro-mechanical post-surface treatment is sandblasting, which is intended to remove the top layer of the resin, making the

glass fibers reachable for chemical interaction. Sandblasting roughens the surface of the post, thus significantly increasing the surface area [10]. The Co-Jet system uses silicate-coated alumina particles and increases the surface area and roughness. A silicate layer is welded onto the post surface. Then, the formed surface can be silane treated to combine the micro-mechanical and chemical mechanisms for surface treatment [11]. Controversial data have been reported when the Co-Jet system was applied, as the surface treatments of the Co-Jet system have been reported to be aggressive [16, 17]. Sahafi et al. [18] evaluated the effectiveness of sandblasting the surface of fiber posts with the Co-Jet system. Satisfactory bond strengths were obtained, although the authors remarked that the treatment was considered too aggressive for the fiber posts. These results may have been influenced by application time, alumina particle size, and pressure [18]. Moreover, Zicari et al. [11] found that Co-Jet sandblasting showed different effects on bond strength in terms of post types. The results of the present study showed that Co-Jet sandblasting enhanced the push-out bond strength. The tribochemical coating of post surfaces, followed by silanization after the Co-Jet procedure, was confirmed by SEM. SEM showed increased micro-retentive surfaces after Co-Jet sandblasting of the fiber-reinforced posts. Similar findings were reported in previous reports in which tribochemical silica coating was demonstrated to enhance the bond strength of composite resin cement to posts [11, 12].

The effect of HF acid etching increased the bond strength of fiber-reinforced post than the control groups. This finding is confirmed by recent results in which 9.5% HF acid was used for surface treatment [19]. The effectiveness of HF acid on fiber post surfaces depends on etching time [20]. In the present study, an etching time of 60 s and a similar HF acid concentration were selected for the surface conditioning of the fiber post according to the previously report [21]. SEM images demonstrated the etching effect on the glass fibers, but conditioning with HF acid seemed to be aggressive and affected not only the methacrylate resin matrix but also the glass fibers, which are components of the FRC post. Despite the increased bond strength after the HF acid treatment, long-term studies are still required as the physico-mechanical properties of the FRC post may be adversely affected. Valandro et al. [21] presented this effect of HF acid on a quartz-fiber post, supporting the present results.

The various laser types and power settings were used for the surface treatment of the FRC post in previous reports [12, 19, 22]. The present study showed that using Er, Cr: YSGG lasers on the post surface increased the bond strength for all power settings used. However, when a lower power setting was chosen (1 W and 2 W), this promising effect was clearly observed and changes in the bond strength values were significant. These results were different from those of Kurt et al. [23] who used the Er: YAG laser with different power settings. The authors stated that all the Er: YAG laser surface treatments groups in their study showed lower bond strengths than the control group. The present study exhibited favorable results of Er, Cr: YSGG laser treatments. Er: YAG and Er, Cr: YSGG lasers generally have similar effects on dental hard tissues and resin-based materials. However, in a previous report, the

Er: YAG laser significantly influenced the push-out bond strength of the FRC post only at higher power settings (4.5 W) [12]. This discrepancy may be due to the structure and design of the FRC post. A methacrylate-based fiber post was used in the present study, whereas their fiber post had an epoxy-based resin matrix. Fiber posts contain a methacrylate or an epoxy-based resin matrix. Unfortunately, the exact composition of the resin matrix is mostly unknown. Moreover, the differences between the present study and the previous report may also result from the power settings of the lasers. In present study, 1 W, 2 W, and 3 W laser power settings and 140 μ s to 200 μ s pulse duration were chosen for the Er, Cr: YSGG laser, but higher power settings and 100 μ s repetition rate were used in previous reports.

The different post types showed different surface topographies, which are expected to significantly influence post-surface characteristic and the micro-mechanical interlocking sites [11]. Moreover, chemical copolymerization between the resin matrix on the surface of the fiber post and the methacrylate-based composite cement or core affects the bonding [11, 24]. However, the fiber post based on epoxy resin copolymerizes less because methacrylate-based composites hardly react chemically with epoxy polymers [25]. Zicari et al. [11] found that the degree to which both micro-mechanical and chemical bonding contribute to the bonding of the post is not clearly known. Micro-mechanical interlocking, which depends on the post-surface topography, is hypothesized to contribute the most to it. According to the SEM images, this hypothesis is confirmed by the present study results. The control group without surface treatment clearly showed smooth a surface of the fiber post, as affirmed by the lowest bond strength. Moreover, the 3 W Er, Cr: YSGG laser treatment showed a scattered fiber post that provides mechanical retention. The SEM images of the Co-Jet sandblasting and lower power settings of the laser treatments at 1 W and 2 W showed that the post surface produced both mechanical retention and chemical interactions, supporting the bond strength results.

Although the results of this study are promising and satisfactory, the bond strengths obtained with different treatment protocols could be considered too aggressive for the FRC post, as they carry the risk of modifying the mechanical properties. Therefore, further studies are needed to evaluate this factor. The current study was limited to one type of fiber-reinforced post and composite resin core build-up material. The extent of the superficial changes on the fiber-reinforced post surfaces depends on both the energy density of the laser irradiation and the type of irradiated FRC post. To achieve better clinical performance, further *in-vivo* studies are required to confirm our findings, and further studies are also necessary to determine whether this increased bond strength is long term or not.

5. Conclusion

Within the limitations of the present study, the surface treatment of the post can affect the bond strength between the methacrylate resin-based FRC post and the core material.

The surface treatments by the 1 W and 2 W Er, Cr: YSGG laser, Co-Jet sandblasting, and HF acid etching enhanced the bond strength between the methacrylate resin-based FRC post and the core material.

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