

## ORIGINAL ARTICLE

## Evaluation of surface roughness and bond strength of quartz fiber posts after various pre-treatments

GULSAH E. AKIN<sup>1</sup>, HAKAN AKIN<sup>2</sup>, CUMHUR SIPAHI<sup>3</sup>, BULENT PISKIN<sup>3</sup> & OMER KIRMALI<sup>4</sup><sup>1</sup>Department of Restorative Dentistry, <sup>2</sup>Department of Prosthodontics, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey, <sup>3</sup>Department of Prosthodontics, Gulhane Military Medical Academy, Ankara, Turkey, and <sup>4</sup>Department of Prosthodontics, Faculty of Dentistry, Akdeniz University, Antalya, Turkey**Abstract**

**Purpose.** Debonding at the post-adhesive interface is a major problem for quartz fiber posts. The objective of this study was to evaluate surface roughness and bond strength of quartz fiber posts after various surface treatments. **Materials and methods.** Sixty-six quartz fiber posts were randomly divided into six experimental groups ( $n = 11$ ) including group C, untreated (control); group SB, sandblasted; group SC, silica coated; group HF, hydrofluoric acid-etched; group N, Nd:YAG laser irradiated; group E, Er:YAG laser irradiated. Surface roughness of the posts was measured before and after pre-treatment. They were then bonded to resin cement and tensile bond strength was determined in a universal testing machine. Furthermore, two-way ANOVA and *post hoc* comparison tests ( $\alpha = 0.05$ ) were performed on all data. **Results.** The highest mean force value was observed in group SB and followed by group E. Tukey's HSD test showed that there was no statistical difference between group SB and group E ( $p = 0.673$ ). The highest mean roughness value was observed in group SB and a significant difference was found between group SB and all other groups ( $p < 0.001$ ). This study reveals that sandblasting and Er:YAG laser irradiation provided a significant increase in bond strength between quartz fiber posts and resin cement. **Conclusions.** Sandblasting or Er:YAG laser-irradiation of the surface of the quartz fiber post before cementation is recommended for increasing retention.

**Key Words:** acid-etch, bond strength, laser, quartz fiber post, sandblasting, silica coating**Introduction**

Pre-fabricated fiber post systems have been widely accepted as a treatment option offering not only esthetics and function [1–4], but also saving time and reducing costs [5–7]. Fiber posts consist of fibers including carbon, glass, quartz and zircon in a resin matrix with a silane coupling agent binding the fibers and matrix together [3]. The number of fibers varies between 25–35, depending on the post type. They are oriented parallel to the post longitudinal axis and their diameter ranges between 6–15  $\mu\text{m}$  per  $\text{mm}^2$  of post cross-sectional surface [8].

An important characteristic of fiber posts is their similar elastic modulus to dentine ( $\approx 20$  GPa), resin cement and resin core materials, producing a stress field similar to that of natural teeth, thus yielding a

protective effect against root fracture [1,3,5–16]. Conversely, titanium, stainless steel and zirconia have elastic moduli (110 GPa, 200 GPa, 210 GPa, respectively) well above that of dentine (18 GPa) [8]. Akkayan and Gulmez [17] reported that teeth restored using quartz fiber posts featured a higher resistance to fracture than those restored using the other three systems including glass fiber, zirconia and titanium posts, whereas both fiber posts resulted in more favorable failure modes (fractures able to be repaired) compared to titanium or zirconia posts. Furthermore, Mannocci et al. [18] researched on intermittent loading of teeth restored with quartz fiber, carbon-quartz fiber and zirconia posts. They found that fiber-reinforced posts were able to reduce to a minimum the risk of root fractures and displayed significantly higher survival rates than teeth restored with zirconia posts.

On the other hand, major concern about the post systems is the lack of durable bond due to debonding at the post–resin interface [2,6,13,14]. The retention obtained from post systems depends on various factors, but the common requirement from all post systems is maximum retention against tensile forces [19]. Thus, to enhance the bond strength of posts to the resin cement, different surface treatments have been proposed, which either mechanically or chemically facilitate the post–resin bonding [3,6,12,13]. Sandblasting with aluminum oxide ( $\text{Al}_2\text{O}_3$ ) particles [3,5,6], etching with hydrofluoric acid (HF) [12], silane application [1,8,20] and silica coating or tribochemical coating with silica modified  $\text{Al}_2\text{O}_3$  particles are commonly used surface treatments [21,22]. Furthermore, recently, lasers have been shown to provide a relatively safe and easy means of altering the surface of materials for improving roughness and bond strength [23–28].

The existing literature does not feature any study that compares the effects of different surface treatment including sandblasting, silica coating, acid etching and lasing on surface roughness of quartz posts and tensile bond strength between quartz posts and resin cement. Therefore, the aim of this *in vitro* study was to investigate effects of different surface treatments including sandblasting, HF, silica coating, Nd:YAG and Er:YAG laser treatments on the strength of the bond between quartz fiber posts and resin cement. It was hypothesized that surface treatment would not significantly affect the bond strength between the quartz fiber posts and resin cement.

## Materials and methods

The study protocol was approved by the local Ethics Committee in Research of the local institution (Cumhuriyet University), protocol 2012-01/09.

Sixty-six quartz fiber posts (DT Light Post, Bisco, Inc., Schaumburg, IL), with a maximum diameter of 1.5 mm in the cylindrical coronal portion and 0.9 mm at the radicular end, were used for this study. Materials and their compositions used in this study are

presented in Table I. Posts were then randomly divided into six experimental groups ( $n = 11$ ), according to the surface treatments performed: control (C), sandblasted (SB), silica coated (SC), hydrofluoric acid-etched (HF), Nd:YAG laser irradiated (N) and Er:YAG laser irradiated (E).

Prior to surface treatments, the surface roughness (Ra) values were measured with a surface roughness profilometer (Mitutoyo surf-tester SJ-401; Mitutoyo Corporation, Kanagawa, Japan) with a diamond stylus (tip radius 5  $\mu\text{m}$ ). A reading was obtained from the needle passing across 0.8 mm length at 1 mm/s to the nearest 0.01  $\mu\text{m}$ . This procedure was repeated one more time in the same position for a total of two readings. Thus, two readings, each consisting of two lengths of 0.8 mm, were made at each position, resulting in a total reading length of 1.6 mm. A final Ra average was calculated and the means of individual specimens were averaged.

- Group C—untreated (control): This group served as the control group, so no treatment was applied to the post surfaces in this group.
- Group SB—sandblasted: Bonding surfaces of post specimens were sandblasted (Ney, Blastmate II, Yucaipa, CA) with 110  $\mu\text{m}$   $\text{Al}_2\text{O}_3$  for 10 s. The air pressure for sandblasting was maintained at 2 bars. Specimens were perpendicularly mounted in a special holder at a distance of 10 mm between the surface of the specimen and the blasting tip. Then, the specimens were rinsed under running water and then dried with oil-free compressed air to remove the remnants for 10 s.
- Group SC—Silica coated: The posts were abraded with an intra-oral airborne-particle-abrasion device (Co-Jet, 3M ESPE, St Paul, MN) using 30  $\mu\text{m}$  silica-modified  $\text{Al}_2\text{O}_3$  particles (CoJet Sand) while rotating the post manually for 10 s. The abrasive was applied perpendicular to the specimens' surfaces at 2 bars pressure from a distance of 10 mm.
- Group HF—Hydrofluoric acid-etched: The posts surfaces were etched with 9.5% hydrofluoric acid (Ultradent Products, Inc., South Jordan, UT) for 20 s.

Table I. Materials and their compositions used in this study.

Materials	Type	Composition	Manufacturer
DT-light post	Translucent quartz fiber post	62% quartz fiber, 38% epoxy resin matrix	Bisco, Inc., Schaumburg, IL
DUO-LINK	Dual-cured composite luting cement	Base: Bis-GMA, TEGDMA, UDMA, glass filler Catalyst: Bis-GMA, TEGDMA, glass fiber	Bisco, Inc., Schaumburg, IL
Rocatec Pre	$\text{Al}_2\text{O}_3$ particles	$\text{Al}_2\text{O}_3$ particles size 110 $\mu\text{m}$	3M ESPE, St Paul, MN
CoJetSand	Tribochemical silica-coating particles	Silica-modified $\text{Al}_2\text{O}_3$ particles of 30 $\mu\text{m}$	3M ESPE, St Paul, MN
Ultradent	Porcelain acid gel	9.5% hydrofluoric acid gel	Ultradent Products, Inc., South Jordan, UT

Bis-GMA, bisphenol glycidyl methacrylate; TEGDMA, triethyleneglycol dimethacrylate; UDMA, urethane dimethacrylate;  $\text{Al}_2\text{O}_3$ , Aluminum oxide.

- Group N—Nd:YAG laser irradiated: Bonding surfaces of post specimens were irradiated by Nd:YAG laser (Smarty A10, Deka Laser, Florence, Italy) with contact, no distance. Laser energy was delivered in pulse mode with a 300  $\mu\text{m}$  in diameter laser optical fiber for 20 s. The laser parameters used were 100 mJ (pulse energy), 10 Hz (repetition rate), 1 W (output power), 300  $\mu\text{s}$  (pulse duration) and 141.54  $\text{J}/\text{cm}^2$  (energy density). Furthermore, air cooling was used during laser irradiating of the specimens.
- Group E—Er:YAG laser irradiated: The bonding surfaces of the post specimens were irradiated with an Er:YAG laser (Smart 2940D Plus, Deka Laser, Firenze, Italy). Laser energy was delivered in pulse mode by a 4 mm diameter titanium articulated arm transmission system with a repetition rate of 10 Hz, energy of 150 mJ, output power of 1.5 W, energy density of 119.42  $\text{J}/\text{cm}^2$  and a pulse duration of 700  $\mu\text{s}$  for 20 s. The distance of application was 10 mm. In addition, water irrigation was used during the lasing of the post specimens.

Following surface treatments, surface roughness of the posts were measured as described before. In addition, representative post specimens were examined under a scanning electron microscope (SEM, LEO 440, Zeiss, Oberkochen, Germany). They were photographed at 1000 $\times$  magnification. SEM analysis was performed with secondary electron (SE) mode under 20 kV accelerating voltage in vacuum ( $3 \times 10^{-4}$  Pa) and 80  $\mu\text{A}$  beam current.

For preparing tensile test specimens, a plastic tube (10 mm height) was used. It has a small central pit in order to obtain a standardized central position of the

post. Prior to cementation procedure, an adhesive resin (ONE-STEP PLUS, Bisco, Inc.) was applied to the posts' surfaces and light cured (Hilux 550; Hilux, Ankara, Turkey) for 10 s according to the manufacturer's instructions. Resin cement is already in a plastic syringe and includes mixing tips, thus equal amounts of paste A and B were dispensed. The resin cement material (DUO-LINK, Bisco, Inc.) was injected into the plastic tube; posts were placed into it and light-cured for 40 s according to the manufacturer's instructions, using a halogen light curing unit (Hilux 550; Hilux). Additional 40 s irradiations were performed from each side of the cylinder prior to the removal of the specimens into the plastic tube to ensure optimal polymerization of the resin material. After removing cemented posts from the plastic tube, they were then placed into a silicone mold and an autopolymerizing acrylic resin material was applied into the mold. Therefore, tensile test specimens ( $n = 10$ ) were obtained using this method (Figure 1).

After the specimens were stored in distilled water at 37°C for 7 days in a water bath machine (BM 402, Nüve, Ankara, Turkey), they were mounted in the custom jig of a universal testing machine (Lloyd LF Plus, Ametek Inc, Lloyd Instruments, Leicester, UK) and subjected to a tensile load parallel to the long axis of the posts at a cross-head speed of 1 mm/min until failure occurred. The tensile bond strength values (N) were calculated from this measurement. Furthermore, the fractured specimens were examined under a stereomicroscope (SMZ 800) at 40 $\times$  magnification to evaluate the fracture pattern and modes of failure were categorized into one of three categories: *adhesive failure* refers to total separation at the interface between the post and resin cement; *cohesive failure* refers to fracture within the post or resin cement; *mixed failure* refers to both. All fracture observations were conducted by one person. The data were analyzed using a two-way ANOVA test. In addition, *post hoc* Tukey comparison tests were performed at a 0.05 significance level.

## Results

Table II summarizes the mean values and standard deviations of tensile bond strength and surface roughness for all groups. Two-way ANOVA revealed significant differences among the different surface treatment groups ( $F = 27.527$  and  $p < 0.001$ ). The highest mean force value was observed in group SB and followed by group E. Moreover, it was observed that there was no statistical difference between group SB and group E ( $p = 0.673$ ). Furthermore, differences between these groups (SB and E) and other groups (C, SC, HF and N) were found to be statistically significant ( $p < 0.001$ ). The lowest mean bond strength value was observed in group N. Analysis of the data also revealed that no significant difference

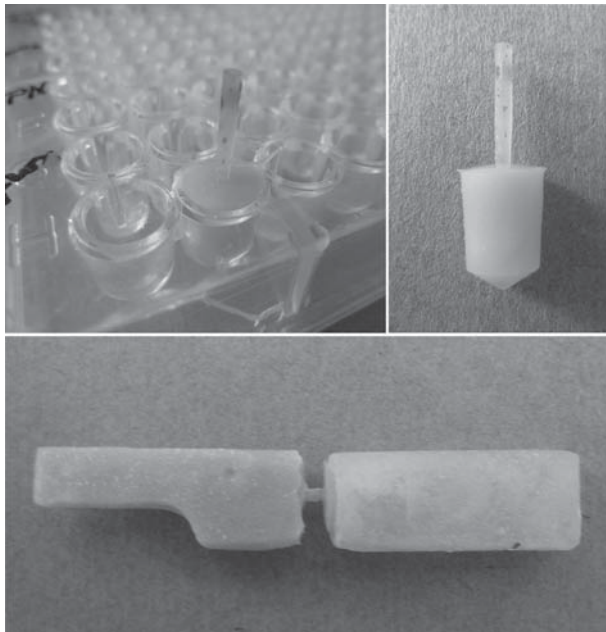


Figure 1. Specimen preparation for tensile bond strength testing.

Table II. Mean tensile bond strength (N) and SD values, surface roughness values (Ra) before and after surface treatments and percentage of failure mode obtained for each tested group.

Groups	Bond strength	Roughness
Group C	318.4 <sup>a</sup> (30.0)	1.1 <sup>A</sup> (0.1)
Group SB	444.4 <sup>b</sup> (53.6)	4.5 <sup>D</sup> (0.6)
Group SC	320.0 <sup>a</sup> (39.9)	2.2 <sup>B</sup> (0.2)
Group HF	309.4 <sup>a</sup> (34.3)	2.4 <sup>BC</sup> (0.2)
Group N	298.2 <sup>a</sup> (27.1)	2.7 <sup>C</sup> (0.2)
Group E	419.1 <sup>b</sup> (37.1)	1.3 <sup>A</sup> (0.1)

For bond strength: values with small letters indicate no statistically significant difference ( $p > 0.05$ ).

For surface roughness: values with capital letters indicate no statistically significant difference ( $p > 0.05$ ).

was found between all other groups (C, SC, HF and N) ( $p > 0.05$ ).

Surface observation of the debonded specimens revealed that most specimens fractured at the interface of the post and resin cement material in groups C, SC, HF and N. Group SB specimens exhibited similar adhesive and cohesive failures. Furthermore, predominant cohesive failure mode was seen in group E specimens (Table III).

On the other hand, two-way ANOVA revealed significant differences among the different surface treatment groups in surface roughness values ( $F = 131.846$  and  $p < 0.001$ ). The highest mean roughness value was observed in group SB and a significant difference was found between group SB and all other groups ( $p < 0.001$ ). In addition, there was a significant difference in roughness between group C and all other groups ( $p < 0.001$ ), except group E ( $p = 0.829$ ). Group SC specimens and group HF specimens showed similar roughness values ( $p = 0.667$ ).

Figures 2(A–F) show the representative specimens of the investigated pre-treatment procedures. Untreated posts exhibited a smooth surface, whereas surfaces of the other treatments showed irregularities. In group SC, the whole surface of the post was coated with silica particles. Moreover, sandblasting resulted in distinctive irregularities on the post surfaces because of fiber damage or ruptures. Similarly,

Table III. Failure type distributions of groups for each specimen.

Groups	<i>n</i>	Adhesive	Cohesive	Mixed
Group C	10	7 (70%)	—	3 (30%)
Group SB	10	4 (40%)	4 (40%)	2 (20%)
Group SC	10	5 (50%)	2 (20%)	3 (30%)
Group HF	10	7 (70%)	—	3 (30%)
Group N	10	6 (60%)	2 (20%)	2 (20%)
Group E	10	3 (30%)	4 (40%)	3 (30%)

both inter-fiber area and fibers of the post were roughened by Nd:YAG laser irradiation. Nevertheless, it can be seen that HF acid affected on inter-fiber area or epoxy resin matrix and there is no difference on fibers. Er:YAG laser irradiation on the post surface resulted in fiber ruptures and it also removed epoxy resin matrix. Thus, spaces were created among fibers.

## Discussion

The use of SB acid and Er:YAG laser as surface pre-treatments improved the interfacial bond strength between quartz fiber post and resin cement material. Nevertheless, the post surface treatments including silica coating, HF acid-etching and Nd:YAG laser irradiation did not affect tensile bond strength. Thus, the null hypothesis was partially accepted. In addition, sandblasted posts exhibited the highest bond strength and roughness values, it was found that there was not any correlation between bond strength and roughness results in all other groups.

Cheleux et al. [29] found that sandblasting increased the bond strength of quartz fiber posts to resin composite materials. Furthermore, Choi et al. [6] demonstrated that surface treatment with sandblasting resulted in significantly higher bond strength of the resin-based luting agent to the quartz fiber post, without any additional treatments. The results of the present study were in accordance with those of Cheleux et al. [29] and Choi et al. [6].

D'Arcangelo et al. [30] reported that the increase in bond strength was more remarkable both in sandblasted and HF acid-etched quartz fiber posts than silanized quartz fiber posts. Therefore, the results of the present study are partially in agreement with those of D'Arcangelo et al. [30]. In addition, Cekic-Nagas et al. [3] recorded that HF acid-etched quartz posts significantly exhibited higher bond strength values than sandblasted and untreated quartz posts to resin core material (Biscore, Bisco, Inc.), whereas both HF acid-etched and sandblasted posts significantly showed higher bond strength values than untreated posts to resin composite material (Admira, Voco, Cuxhaven, Germany). Furthermore, Valandro et al. [31] stated that silica coating improved the bond strength between quartz fiber posts and resin cements when compared with phosphoric acid or HF acid-etching. Thus, the results of the present study contradicted those of Cekic-Nagas et al. [3] and Valandro et al. [31].

Surface treatment of the quartz posts with different lasers is being described for the first time in this study. Different lasers exhibited different results in the present study. Er:YAG laser irradiated posts demonstrated significantly higher bond strength than Nd:YAG laser irradiated posts. It can be explained that Nd:YAG laser application resulted in larger pits than Er:YAG laser application on the surfaces of the posts.

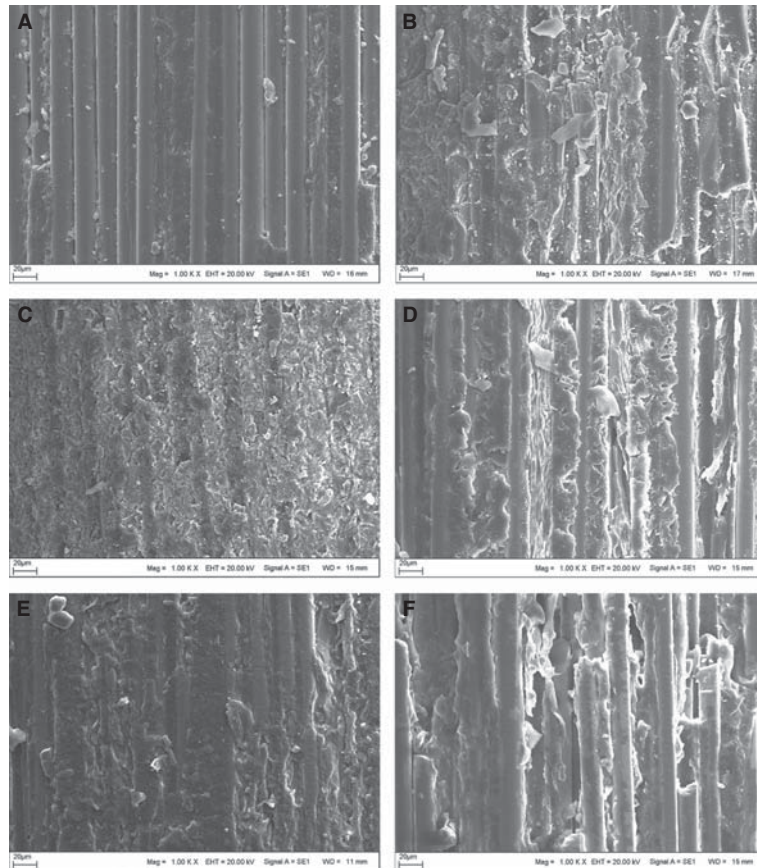


Figure 2. Representative SEM micrographs of the quartz fiber posts (original magnification: 1000 $\times$ ). (A) Untreated post, (B) sandblasted post, (C) silica coated post, (D) HF acid-etched post, (E) Nd:YAG laser irradiated post, (F) Er:YAG laser irradiated post.

There is only one study on Er:YAG laser pretreatment of quartz post in the existing literature [32]. Contrary to results with the present study, Tuncdemir et al. [32] reported that both Er:YAG laser irradiation and sandblasting did not affect bonding of the post. Most reports [9,33] evaluated adhesion between posts and laser irradiated root canal dentin in the literature. Both for Nd:YAG and Er:YAG laser applications, many different experimental set-ups have been used in the literature and application of lasers in different parameters such as energy, output power and pulse duration can affect the results of the studies. Hence, to determine the laser parameters used in this study, a pilot study was carried out. Based on those results and previous study [23], 100 mJ, 10 Hz and 1 W for Nd:YAG laser and 150 mJ, 10 Hz and 1.5 W for Er:YAG laser were selected. In addition, the distance of the laser application was selected based on the previous studies [23-28]. Future investigations could focus on which parameters are more suitable for bond strength of the fiber posts.

The highest mean roughness value was observed in sandblasted posts the same as bond strength value. However, although other surface treatments (except Er:YAG laser) significantly enhanced the surface roughness just as sandblasting, they were ineffective

in improving adhesion of post to resin cement material. In addition, despite the fact that Er:YAG laser irradiated posts exhibited the same roughness values with untreated posts, they had one of the highest tensile bond strength values. Thus, it was found that there was no correlation between bond strength and surface roughness results.

The failure mode registered in the all tested groups was predominantly adhesive except group SB and group E. Similar adhesive and cohesive failure modes were seen in group SB (40%), whereas predominantly cohesive failure mode was seen in group E (40%). Therefore, there was a correlation between tensile bond strength and failure types.

On the other hand, the DT Light-Post system used in this study, which contains epoxy resin as the matrix connecting the individual fibers, has no functional groups to react with a silane coupling agent. It was previously reported that silane does not bond well with the epoxy matrix [2]. Similarly, consistent with the present study, Choi et al. [6] stated that silane treatment did not have a significant effect on the shear bond strength of the posts.

When evaluating SEM micrographs, it can be seen that there are fiber ruptures in the sandblasted specimen. Moreover, Er:YAG laser irradiation resulted in separation among fibers by removing inter-fiber area.

Thus, cavities occurred among fibers and it could be expected that resin cement could penetrate into these cavities. Er:YAG laser irradiation significantly affected the tensile bond strength of glass fiber posts to resin cement. Nevertheless, this damage both on fibers and inter-fiber area could impair structure of the post. Although sandblasting resulted in more gentle damage than Er:YAG laser irradiation, future investigations could focus on fracture strength of the fiber posts after sandblasting and Er:YAG laser irradiation.

One of the limitations of this study was that *in vivo* conditions were not simulated. Cyclic loading as well as human chewing patterns and thermocycling effect were not evaluated in the present study. Although these storage types are important to simulate clinical conditions, the results of this study may be beneficial for the comparison of the effects of different surface treatment methods.

## Conclusions

Within the limitations of this *in vitro* study the following conclusions were drawn:

- (1) Surface treatments with sandblasting and Er:YAG laser irradiation were found to be effective methods for improving the bonding of quartz fiber posts to resin cement.
- (2) No correlation was found between bond strength and surface roughness results.

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