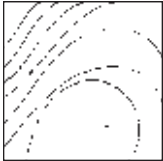


Effect of Various Surface Treatments on the Bond Strength of Porcelain Repair



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This study evaluated the effect of surface treatments on the repair strength of composite resin on a feldspathic ceramic. Ninety ceramic specimens were divided into six groups. In the experimental groups, 4% hydrofluoric acid etching, Er:YAG laser irradiation, CO₂ laser irradiation, airborne-particle abrasion, and silica coating were used as surface treatments. After the application of a porcelain repair kit, composite resin was placed on the treated surfaces. After a shear bond strength test, data were statistically analyzed ($\alpha = .05$). Surface treatments increased the repair bond strength values ($P < .05$). Airborne-particle abrasion and silica coating were found to be the most effective. CO₂ laser showed higher repair strength values than Er:YAG laser. (Int J Periodontics Restorative Dent 2013;33:e120–e126. doi: 10.11607/prd.1362)

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In recent years, core-veneered all-ceramic restorative materials have gained popularity in dentistry.¹ The combination of the high strength of ceramic cores and the superior esthetics of weaker veneering ceramics results in reliable and more biocompatible restorations.² Among all-ceramic materials, the use of zirconium-oxide ceramic has increased due to its high flexural strength, transformation toughening mechanism, white appearance, and chemical and structural stability.^{3,4} While an acceptable esthetic appearance is obtained by adding veneering ceramics in layers to the zirconium-oxide ceramic core, the interface between the core and veneering material is a problem as it may cause ceramic chipping or cracking.⁵ Fracture and chipping caused by sliding contact continue to be the most common failure modes for zirconia-based ceramic restorations.⁶ Previous studies found the bond strength of veneering ceramics to zirconium oxide to be higher than the shear bond strength of veneering ceramic itself. Results showed that failures that occurred between the veneering material and zirconium-oxide

ceramic were not adhesive but cohesive fractures in the veneering ceramic near the interface, with residual veneering material remaining on the zirconia surface.^{7,8}

Fracture of a ceramic restoration generally occurs after cementation because of masticatory forces, change of temperatures, saliva, and pH changes.⁹ Because of the common use of adhesive cementation, it is impossible to remove the fractured restoration without trauma to the restored tooth. Additionally, this procedure is time consuming and costly.^{10,11} Consequently, intraoral repair of the fractured restoration with composite resin restorative material is a quick and simple procedure with minimal expense, but has disadvantages such as staining, poor wear resistance, and weak bond strength to the restoration.¹² To increase the bond strength of composite resin material to fractured ceramic, various surface treatments are used, such as acid etching, airborne-particle abrasion with aluminum oxide (Al₂O₃) particles¹³ or Al₂O₃ particles modified with silica,^{14,15} and, in recent years, roughening the ceramic surface using laser irradiation.^{16–20}

The use of lasers in dentistry has gained popularity in recent years. For intraoral soft tissue surgery and hard tissue applications, carbon dioxide (CO₂) and neodymium-doped yttrium aluminum garnet (Nd:YAG) lasers are the most preferred instruments.¹⁶ Additionally, laser applications are used as a ceramic surface treatment to create surface roughness and obtain

more surface area for increasing the bond strength of the resin cement.^{16,21} The CO₂ laser is suitable for the treatment of ceramic materials because of its emission wavelength.¹⁶

The erbium:yttrium-alumina garnet (Er:YAG) laser has been used for different purposes in clinical dentistry, such as cavity preparation, surface conditioning, or carious dentin removal.^{19,20} Additionally, Er:YAG laser irradiation is the preferred surface treatment for dental ceramics due to its wavelength coinciding with the main absorption peak of water and is well absorbed by hydroxide ion (OH⁻) groups in hydroxyapatite.²²

The objective of the present study was to evaluate the effect of different surface treatments on the repair strength of composite resin on veneering feldspathic ceramics of zirconia. The hypothesis was that surface treatments will promote different bond strengths.

Method and materials

Ninety disk-shaped (10 mm in diameter and 2-mm thick) zirconium-oxide core specimens were produced using a copy milling system (Zirconzahn) with prefabricated blanks of zirconium oxide (ICE Zircon Translucent, Zirconzahn) and then sintered according to manufacturer instructions. One of the flat surfaces of the sintered specimens was veneered with feldspathic ceramic (VITA Zahnfabrik) at a 2-mm thickness using a manual layering technique. Specimens were then

embedded in autopolymerizing acrylic resin blocks (Meliodent, Heraeus Kulzer) with veneering ceramic facing upwards. Veneered ceramic surfaces were ground with 600-grit silicon carbide paper (Carbimet, Buehler) and then ultrasonically cleaned in distilled water for 3 minutes. Subsequently, specimens were divided into six groups of 15 specimens for the following surface treatments: Group C (control), no surface treatment; group H, specimens were etched with 4% hydrofluoric acid (Bisco) for 5 minutes according to manufacturer instructions; group EL, an Er:YAG laser (Fotona AT Fidelis, Fotona) was used to irradiate the ceramic surfaces at a power output of 2 W (200 mJ/pulse 10 Hz) for 10 seconds; group CL, the ceramic surfaces were irradiated using a CO₂ laser (Smart US20D, Deka) (laser energy was delivered in a pulse mode with a wavelength of 10.6 μm, a pulse repetition rate 1,000 Hz, and a pulse duration of 160 ms at a mean power setting of 3 W); group AA, airborne-particle abrasion with 50-μm Al₂O₃ particles using an intraoral device (Microetcher, Danville) at a distance of approximately 10 mm and a pressure of 2.5 bars for 15 seconds; and group SC, the ceramic surfaces were treated with 30-μm Al₂O₃ particles modified by silica (Cojet Sand, 3M ESPE) with a nozzle distance of approximately 10 mm for 20 seconds.

After surface treatments, two coats of prehydrolyzed silane primer (Porcelain Primer, Bisco) were applied to the treated surfaces, allowed to soak for 30 seconds,

and then dried with an air syringe. Next, one coat of one-step adhesive (One-Step Plus, Bisco) was applied, and, after air drying, the surfaces were polymerized with a light-curing unit (Hilux 200, Benlioglu Dental) with an output of 600 mW/cm² for 10 seconds. Subsequently, a nanohybrid composite resin (Reflexions, Bisco) was placed on the surfaces using a plastic transparent mold with a hole at the center (5 mm in diameter and 2 mm in height). Composite resin was incrementally condensed into the mold until the mold was full, and each layer was light polymerized for 20 seconds at a distance of 1 mm. All specimens were stored in distilled water at 37°C ± 2°C for 24 hours and then thermocycled for 500 cycles between 5°C ± 2°C and 55°C ± 2°C with a dwell time of 30 seconds.

The shear bond test was performed with a universal testing device (Lloyd LRX, Lloyd Instruments) at a crosshead speed of 1 mm/min. The shear bond strength values were calculated in MPa by dividing the failure load (N) by the area of the composite resin ($N/\pi r^2$). Data were statistically analyzed, and the Kolmogorov-Smirnov test showed that the data were of a normal distribution ($P > .05$). One-way analysis of variance (ANOVA) (SPSS version 12.0, SPSS, IBM) was used to determine the significant differences among surface treatments and primers and their interactions. All treatment combination mean repair strength values were compared using the Tukey multiple comparison test ($\alpha = .05$).

In addition, the type of failure was observed by using a stereomicroscope (Stemi 2000-C, Carl Zeiss) at a magnification of ×10 to determine the amount of repair material left behind and to assess any damage to the veneering ceramic that may have occurred during the shear bond test. The nature of failure was noted as adhesive, cohesive, or mixed. Cohesive failure was deemed to have occurred if more than 50% of the testing surface of the ceramic had fractured.

To evaluate the effect of treatments on the ceramic surface, six additional specimens were prepared with the same surface treatment protocols. Next, all specimens were gold sputtered with a sputter coater (s150b, Edwards) and examined under a field emission scanning electron microscope (SEM) (JSM-6335F, Jeol) at 20.0 kV. The SEM photomicrographs were taken at ×500 magnification for visual inspection.

Results

The results of one-way ANOVA are shown in Table 1. Significant differences were found between the surface treatments ($P < .05$). The repair strength of composite resin to veneering ceramic depended on the surface treatment method. The mean repair strength values and standard deviations for each group are presented in Table 2. The lowest repair strength values were obtained with the control group (5.56 MPa) and were found to be signifi-

cantly different from the other groups ($P < .05$). The highest values were obtained with groups SC (17.73 MPa) and AA (16.88 MPa) ($P < .05$). No significant difference was found between groups H (13.71 MPa) and CL (12.99 MPa) ($P > .05$). When the laser-irradiated groups were compared, a significant difference was found between group CL (12.99 MPa) and group EL (10.73 MPa) ($P > .05$). Laser-irradiated groups showed lower repair bond strength values than group H.

The modes of failure are given in Table 3. All specimens from each group, except the control group, showed cohesive failures. In group C, adhesive and mixed failures were observed.

SEM photomicrographs of surface-treated specimens are presented in Fig 1. Surface treatments modified the surface topography of veneering ceramic by increasing the surface irregularities. Minimal irregularities were seen on the surface of the specimen that had no treatment (Fig 1a). Surface defects and irregularities were seen on the photomicrographs of the specimen irradiated with the Er:YAG laser (Fig 1b). More retentive irregularities were seen with the specimens treated with either the CO₂ laser or hydrofluoric acid (Figs 1c and 1d). On the surface of the specimens treated with silica coating and airborne-particle abrasion, more microirregular and rougher surfaces were seen compared with the other surface treatments (Figs 1e and 1f).

Discussion

The results of this study confirmed that surface treatments increased the repair strength of composite resin to feldspathic veneering ceramic of zirconia ($P < .05$). The lowest repair strength values were obtained with the specimens that had no surface treatment. The results showed that although silane application is necessary, especially for feldspathic ceramic, the mechanical locking obtained with the irregularities created on the ceramic surface is the major factor for a strong bond. Previous studies have shown similar results.^{23,24} The control group, which was flattened using silicon carbide paper and received the application of a silane agent, showed low bond strengths (4.15 MPa) that were insufficient for promoting reliable adhesion to the resin cement.²²

In this study, the highest bond strength values were obtained with the specimens treated with 30- μm Al_2O_3 particles modified by silica (17.73 MPa) and 50- μm Al_2O_3 particles (16.88 MPa). No statistical differences were found between the two treatments ($P > .05$). Past studies have stated that different ceramic specimens that received silica coating had similar or higher bond strength than specimens treated with airborne-particle abrasion.^{25,26} The layer left by silica coating provides a basis for silane. At the ceramic-composite bond, silane acts as a coupling agent that adsorbs onto and alters the ceramic surface while increasing bond strength.²⁵ On the other hand,

Table 1 One-way ANOVA results

	Sum of squares	df	Mean square	F	P
Between groups	1475.74	5	259.15	296.62	.0001
Within groups	83.58	84	.995		
Total	1559.33	89			

ANOVA = analysis of variance; df = degrees of freedom; F = frequency distribution.

Table 2 Mean (SD) repair strength values for each group (n = 15)

Group	Mean (SD)*
C (control)	5.56 (0.40)
H	13.71 (0.48) ^a
EL	10.73 (0.90)
CL	12.99 (1.06) ^a
AA	16.88 (1.18) ^b
SC	17.73 (1.50) ^b

*The same letters indicate groups that were not statistically different. SD = standard deviation.

Table 3 Modes of failure by group

Group	Type of failure		
	Adhesive	Cohesive	Mixed
C (control)	13	–	2
H	–	15	–
EL	–	15	–
CL	–	15	–
AA	–	15	–
SC	–	15	–

airborne-particle abrasion increases the surface area and surface energy for adhesion of composite

resin and also creates micromechanical locking. By decreasing the surface tension, airborne-particle

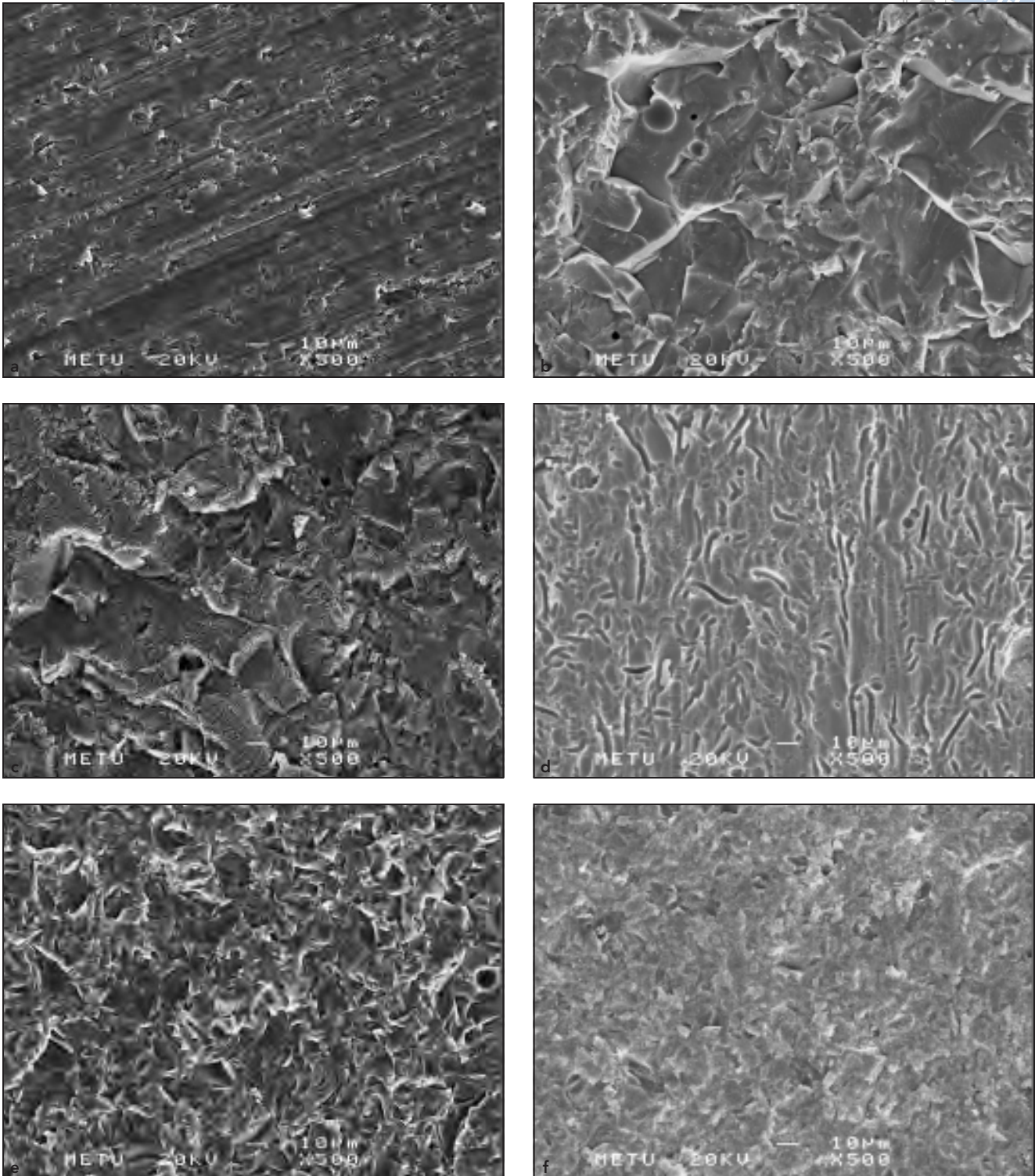
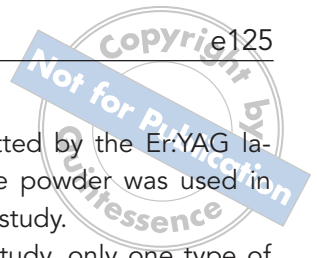


Fig 1 SEM images of the feldspathic veneering ceramic after different surface treatments. (a) Control; (b) irradiation with Er:YAG laser; (c) irradiation with CO₂ laser; (d) etching with 4% hydrofluoric acid; (e) air abrasion with 50- μ m Al₂O₃ particles; and (f) silica coating with 30- μ m Al₂O₃ particles modified by silica.



abrasion enables optimal wetting of silane.¹⁵ However, silica coating does not reduce the ceramic volume and strength as airborne-particle abrasion does.^{26,27}

Hydrofluoric acid etching is one of the most commonly used chemical surface treatments for ceramics.^{22,27} Also, it is a suggested method for ceramic surface etching before the use of repair procedures. In previous studies, it was stated that hydrofluoric acid etching preceding silane application was the most effective ceramic surface treatment, although it was not always statistically different from other treatments.^{28,29} In this study, hydrofluoric acid etching preceding silane application showed higher strength values (13.71 MPa) following silica coating and airborne-particle abrasion, but no differences were found between the two. Similar results were found in studies investigating the effect of surface treatments on the bond strength of composite resin to feldspathic ceramic.^{30,31} In these studies, acid etching showed higher bond strength values than laser irradiation. However, although it creates good bond strength between the feldspathic veneering ceramic and composite resin, it is well recognized as having hazardous effects *in vivo*. So this method is not useful practically in dentistry.²²

In the present study, two different lasers, CO₂ and Er:YAG, were used to irradiate ceramic surfaces. Both increased the bond strength of the composite resin to ceramic when compared with untreated specimens. However, they had

lower bond strength values than silica coating, airborne-particle abrasion, and acid etching. When the effects of two laser treatments on the bond strength of composite resin to ceramic were compared, CO₂ laser treatment showed higher repair bond strength values than Er:YAG laser treatment. Studies have shown that a CO₂ laser is well suited for the treatment of porcelain materials due to its emission wavelength, which is almost totally absorbed by porcelain.³²

Contrasting information exists regarding the effect of Er:YAG laser irradiation as a surface treatment of ceramics on the bond strength of composite resin to ceramic. While Akyıl et al²⁰ found that treating the ceramic surface using an Er:YAG laser (200 mJ/pulse, 10 Hz, 10 seconds) increased the bond strength of resin cement to yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) ceramic, Stübinger et al³³ stated that it was not effective on Y-TZP surfaces at a power output of 10 W. In another study investigating the effect of feldspathic ceramic surface treatment on the bond strength to resin cement, Er:YAG laser irradiation at 500 mJ/pulse, 4 Hz for 2 minutes with 2.95- μ m wavelength showed lower bond strength than untreated specimens.²² In this study, the laser was used at 2 W (200 mJ/pulse 10 Hz) for 10 seconds and showed higher bond strength values than the control group. These conflicting conclusions may be due to the different composition of ceramic materials and its reflectance. To increase the absorption of the

energy emitted by the Er:YAG laser, graphite powder was used in the present study.

In this study, only one type of porcelain repair kit and composite resin were used. For laser treatments, the output power was chosen from the literature that resulted in higher bond strength values. Also, surface roughness and volume loss from the veneering ceramic surfaces were not evaluated. For this reason, clinical studies are necessary to evaluate the long-term behavior of repair strength of composite resin to veneering feldspathic ceramic.

Conclusion

Within the limitations of this study the following conclusions were drawn: (1) surface treatments affected the repair bond strength of a composite resin to veneering feldspathic ceramic of zirconia ($P > .05$); (2) silica coating and airborne-particle abrasion are the most effective methods for increasing repair bond strength and showed higher repair bond strength values; and (3) when laser irradiation is compared with ceramic surface treatment, the CO₂ laser was found to be more effective than the Er:YAG laser.

Acknowledgment

The authors reported no conflicts of interest related to this study.

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