

Effect of Er,Cr:YSGG Laser, Air Abrasion, and Silane Application on Repaired Shear Bond Strength of Composites

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Clinical Relevance

Use of Er,Cr:YSGG (erbium, chromium: yttrium-scandium-gallium-garnet) laser does not improve the shear bond strength of the repaired resin; however, air abrasion with 50- μ m aluminum oxide particles results in higher shear repair bond strength, and therefore is recommended prior to repair. Application of a silane coupling agent does not improve the shear bond strength of the repaired resin.

SUMMARY

Aged resin composites have a limited number of carbon-carbon double bonds to adhere to a new layer of resin. Study objectives were to 1) evaluate various surface treatments on re-

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paired shear bond strength between aged and new resin composites and 2) to assess the influence of a silane coupling agent after surface treatments.

Methods: Eighty disk-shape resin composite specimens were fabricated and thermocycled 5000 times prior to surface treatment. Specimens were randomly assigned to one of the three surface treatment groups (n=20): 1) air abrasion with 50- μ m aluminum oxide, 2) tribochemical silica coating (CoJet), or 3) Er,Cr:YSGG (erbium, chromium: yttrium-scandium-gallium-garnet) laser or to a no-treatment control group (n=20). Specimens were etched with 35% phosphoric acid, rinsed, and dried. Each group was divided into two subgroups (n=10): A) no silanization and B) with silanization. The adhesive agent was applied and new resin composite was bonded

to each conditioned surface. Shear bond strength was evaluated and data analyzed using two-way analysis of variance (ANOVA).

Results: Air abrasion with 50- μm aluminum oxide showed significantly higher repair bond strength than the Er,Cr:YSGG laser and control groups. Air abrasion with 50- μm aluminum oxide was not significantly different from tribochemical silica coating. Tribochemical silica coating had significantly higher repair bond strength than Er,Cr:YSGG laser and the control. Er,Cr:YSGG laser and the control did not have significantly different repair bond strengths. Silanization had no influence on repair bond strength for any of the surface treatment methods.

Conclusion: Air abrasion with 50- μm aluminum oxide and tribochemical silica followed by the application of bonding agent provided the highest repair shear bond strength values, suggesting that they might be adequate methods to improve the quality of repairs of resin composites.

INTRODUCTION

Resin composite is a widely used material in direct restorative procedures. After a period of service, most resin restorations develop defects resulting from wear, fracture, or discoloration. The replacement of resin restorations has become a common restorative procedure.¹ When resin composites are replaced, tooth structure is often removed to expose an etchable substrate. This removal of additional structure results in weakening of the tooth. Resin repair instead of replacement is more conservative and can increase the longevity of restorations at a lower cost.²⁻⁴ A study found that, within two years, the repaired margins showed good results according to the Modified Ryge USPHS Clinical Criteria.⁵ Repair should always be considered before replacement.⁶ Several techniques using mechanical and/or chemical treatments have been proposed to improve bonding. Examples of mechanical treatments to improve bonding are bur abrasion and air abrasion, while examples of chemical treatments are tribochemical silica coating with 30 μm aluminum oxide, hydrofluoric acid, phosphoric acid, silane application, and application of a bonding agent.

The objectives of this study were to explore whether Er,Cr:YSGG (erbium, chromium: yttrium-scandium-gallium-garnet) laser, air abrasion with 50 μm aluminum oxide, a tribochemical silica coating of

30 μm aluminum oxide, or the application of silane improved the repair shear bond strength when compared with adhesive bonding to nontreated surfaces.

MATERIALS AND METHODS

Eighty 16-mm diameter plastic molds were poured with self-cured acrylic resin (Bosworth Fastray, Bosworth Co, Skokie, IL, USA). An 8-mm diameter and 2-mm deep cavity was made in each mold. Retention was created at the bottom of the cavity using an inverted cone bur. Each acrylic surface was polished sequentially with 240, 320, 400, and 600 silicon carbide paper with running water. A micro-hybrid resin matrix composite (Point 4, shade A4, Kerr, Orange, CA, USA) was placed into the preparation using a composite dispensing gun. The top surface of each specimen was pressed perpendicular to the long axis of the cylinder with a Mylar strip and glass slide to obtain a flat surface and remove excess material. The glass slide was removed and the specimens were cured with a light-emitting diode (LED) visible light curing unit (L.E. Demetron I, Kerr) for 40 seconds. The light tip was kept perpendicular to and in contact with the Mylar strip surface to receive maximum curing depth. The light intensity was monitored with a visible light meter (CureRite, Dentsply, Dillford, DE, USA) to verify the light output of $>900 \text{ mW/cm}^2$. All specimens were finished and polished with the same sequence of silicon carbide paper under running water as mentioned above to remove the excess resin and establish the surface perpendicular to the long axis of the specimen.

Aging was simulated on all specimens by thermocycling 5000 times between 6°C and 51°C ($\pm 2^\circ\text{C}$) with a 30-second dwell time and 10-second transfer time.

All specimens were randomly assigned into four major groups. The air abrasion groups were prepared with an intraoral air abrasion unit (Micro-etcher II, Danville Materials, San Ramon, CA, USA) with the same settings, but different size and type of particles.

Group A50: Air Abrasion with Aluminum Oxide 50 μm Particles (n=20)

Air abrasion was performed with an intraoral air abrasion unit using 50- μm aluminum oxide (Al_2O_3) particles (Danville Materials, San Ramon). A uniformly prepared surface was created by moving the handpiece slowly in a circular motion perpendicular

to the specimen surface at a distance of approximately 5 mm, with 60 psi for 10 seconds. Specimens were rinsed with water for 10 seconds and dried with an air syringe at a distance of 10 mm for 5 seconds.

Standardization for distance between the tip and specimen was made using stone as a base for securing the specimen and a plastic cone with resin composite as a parallel guide on the top. An analyzing rod and surveyor were used to ensure a perpendicular approach to the specimen surface.

Group ATS: Air Abrasion with Tribochemical Silica Coating (n=20)

The specimens were air abraded using 30- μm Al_2O_3 particles that were modified with silica (CoJet system, 3M ESPE, St Paul, MN, USA). The specimen preparation was the same as for group A50.

Group ECL: Er,Cr:YSGG Laser (n=20)

Laser preparation was performed with an Er,Cr:YSGG laser device (Waterlase, Biolase Technology, San Clemente, CA, USA) in a noncontact and focused mode. The specimen and a stabilizing jig were moved slowly in a circular motion at a distance of approximately 1 mm from the cylinder fiber tip for 10 seconds. The handpiece was adjusted so that the fiber tip irradiated at a 1-mm distance perpendicular to the specimen surface and was secured in silicone connected to a stone jig. The laser parameters were set at a wavelength of 2.78 μm , a pulse frequency of 20 Hz, a pulse duration of 140 μs , a power of 4.0 W, an air pressure setting of 65%, and a water pressure setting of 55%.

Group NST: No Surface Treatment (n=20)

After the surface treatments were completed, the specimens and the control were divided into two subgroups each. The specimens in groups A50-A, ATS-A, ECL-A, and NST-A were not treated with the silane coupling agent. The specimens in groups A50-B, ATS-B, ECL-B, and NST-B were further treated with a silane coupling agent (Silane, Ultradent Products, South Jordan, UT, USA) for 60 seconds and air dried. All groups were then etched with 35% phosphoric acid (Ultra-Etch, Ultradent Products) for 15 seconds. The specimens were rinsed with water for 10 seconds and dried with air for 5 seconds.

Two consecutive layers of the adhesive agent (OptiBond Solo Plus, Kerr) were applied on all specimens. The solvent was evaporated using a gentle stream of compressed air and then the adhesive light cured for 20 seconds.

Cylinders of approximately 4 mm in diameter and 2 mm in height of resin composite were fabricated using a plastic mold and bonded onto the previously treated specimens. After bonding, the specimens were stored in distilled water at 37°C for 24 hours.

After recording the diameters, all specimens were secured in a special holder in a universal testing machine (MTS Sintech Renew 1123, Eden Prairie, MN, USA). A stainless steel ring with an inner circular knife-edge was adapted at the interface of the aged and new resin matrix composite surface. Shear load was applied with a crosshead speed of 1 mm/min until fracture with the direction of force perpendicular to the long axis of specimens. The force was calculated in megapascals (MPa).

The specimen sections were examined for type of failure under a stereomicroscope (Prior Scientific Inc, Rockland, MA, USA) at 10 \times and 15 \times magnification to determine the type of failure.

Twelve additional resin composite specimens were prepared and aged by thermocycling as described previously. Then, they were assigned to three surface treatment groups and one control group (n=3) and treated using the group conditions described above. After the surface treatments, all specimens were cleaned with 35% phosphoric acid, rinsed, and dried. Surface roughness testing was performed with a profilometer (Surtronic 3⁺, Taylor Hobson, Leicester, UK). Each specimen was measured six times (4-mm distance) at six different locations. The average surface roughness (Ra) value from each specimen was recorded.

The specimens used for the surface roughness experiment were mounted on a specimen holder with electrically conductive double-sided sticky tape and observed with a scanning electron microscope (SEM; JEOL, JSM-5310LV, Tokyo, Japan). The SEM was operated with an accelerating voltage of 10 kV using both secondary and backscattered electron detectors for imaging. Micrographs were taken at 750 \times magnification in order to document the surface texture created by the different surface treatments after etching with 35% phosphoric acid.

Statistical Analysis

A two-way analysis of variance (ANOVA) model was used to assess the impact of surface conditioning (four levels), silanization (yes or no), and their interaction on peak stress. Pair-wise comparisons were adjusted for multiple comparisons using the Tukey multiple comparisons procedure to control the overall significance level at 5%.

Mantel-Haenszel chi-square tests for ordered categorical responses were used to compare the groups for differences based on failure mode. Pair-wise comparisons were adjusted for multiple testing using the Sidak method; *p* values are presented with and without multiple comparisons adjustment.

RESULTS

Shear Bond Strength

A50 specimens without silane application produced the highest shear bond strength of 19.7 ± 1.1 MPa, followed by A50 specimens with silane application (18.8 ± 1.3 MPa). The results from ATS groups were not statistically different from both A50 groups (18.7 ± 1.3 MPa in ATS without silane application and 17.4 ± 1.4 MPa in ATS with silane application). The shear bond strength of the NST group with silane application was 15.8 ± 1.5 MPa which was not statistically different from the results of both ATS groups. The lowest shear bond strength values were from the ECL groups (14.2 ± 1.4 MPa with silane application and 14.4 ± 1.5 MPa without silane application). The NST without silane application produced a shear bond strength of 14.6 ± 1.1 MPa which was not statistically different from both ECL groups. (Table 1)

A50 and ATS had significantly higher peak stress than ECL without silanization (*p*<0.0001) and with silanization(*p*=0.0002) and higher peak stress than NST without silanization (*p*<0.00001) and with silanization (*p*<0.00001). A50 did not have significantly different peak stress than ATS without silanization (*p*=0.70) or with silanization (*p*=0.33). ATS had significantly higher peak stress than ECL without silanization (*p*<0.0001) and with silanization (*p*<0.0001) and significantly higher peak stress than NST without silanization (*p*<0.0001) but not with silanization (*p*=0.16). ECL and NST did not have significantly different peak stress without silanization (*p*=1.00) or with silanization (*p*=0.11). There was no effect of silanization on peak stress overall (*p*=0.34) for any of the surface conditioning methods (*p*=0.76 for A50, *p*=0.39 for ATS, *p*=1.00 for ECL, *p*=0.39 for NST).

Type of Failure

A50 with and without silanization had significantly higher cohesive failure than ECL with silanization (*p*=0.0139) and without silanization (*p*=0.0111). A50 did not have significantly different cohesive failure than ATS with silanization (*p*=0.99) or without silanization (*p*=1.00) and did not have significantly

Surface Treatments	With Silane Application, MPa	Without Silane Application, MPa
Air abrasion (A50)	18.8 ± 1.3 ^a	19.7 ± 1.1 ^a
Tribochemical silica coating (ATS)	17.4 ± 1.4 ^{a,c}	18.7 ± 1.3 ^{a,c}
Er,Cr:YSGG laser (ECL)	14.2 ± 1.4 ^b	14.4 ± 1.5 ^b
No surface treatment (NST)	15.8 ± 1.5 ^{b,c}	14.6 ± 1.1 ^b

^a The Values Designated With the Same Superscripted Letters Are Not Significantly Different

different cohesive failure than NST with silanization (*p*=1.00) or without silanization (*p*=0.86). ATS without silanization had significantly higher cohesive failure than ECL with silanization (*p*=0.0139) or without silanization (*p*=0.0111). ATS without silanization did not have significantly different cohesive failure than ATS with silanization (*p*=0.99) or NST with silanization (*p*=1.00) or without silanization (*p*=0.86). ATS with silanization had marginally higher cohesive failure than ECL with silanization (*p*=0.06) and without silanization (*p*=0.06). ATS with silanization did not have significantly different cohesive failure than NST with silanization (*p*=0.99) or without silanization (*p*=1.00). ECL without silanization had significantly lower cohesive failure than NST with silanization (*p*=0.0111). ECL without silanization did not have significantly different cohesive failure than ECL with silanization (*p*=1.00) or NST without silanization (*p*=0.13). ECL with silanization had significantly lower cohesive failure than NST with silanization (*p*=0.0139). (Table 2)

Surface Roughness

Surface roughness was analyzed after a log-transformation due to the assumptions required for the ANOVA. All four surface treatments had significantly different surface roughness: ECL > A50 > ATS > NST. P-values for the comparisons were all 0.0001 except for ATS vs NST which had a *p*-value of 0.0019. A50 specimens produced a roughened and irregular surface with numerous microretentive fissures (Figure 1). ATS specimens also created a roughened and irregular surface with noticeably

Groups	Surface Treatment	Cohesive Failure	Mixed Failure	Adhesive Failure
A50-A	Air abrasion without silane application	10	0	0
A50-B	Air abrasion with silane application	10	0	0
ATS-A	Tribochemical silica coating without silane application	10	0	0
ATS-B	Tribochemical silica coating with silane application	8	2	0
ECL-A	Er,Cr:YSGG laser without silane application	1	6	3
ECL-B	Er,Cr:YSGG laser with silane application	1	4	4
NST-A	No treatment without silane application	7	3	0
NST-B	No treatment with silane application	10	0	0

Note: One specimen was lost during the test for group ECL-B

fewer microretentive fissures (Figure 2). ECL specimens had the most noticeable asperities and cracks (Figures 3 and 4). The resin composite surface in the NST group showed a uniformly smooth surface (Figure 5). (Table 3)

DISCUSSION

This study was conducted to examine the effect of different surface treatments on the repair bond strength of aged resin composite. Shear bond

strength was tested because it provides a common measurement of the maximum stress possible at the bonding interface. This method would be most useful in predicting the success of repairs in the anterior region, where restorations are commonly subjected to shear forces during function.

Many studies compare repair bond strength with the cohesive strength of intact resin composite,^{7–10} whereas others set the control as no surface treatment before repairing, to compare with other test groups.^{12–15} Since the bond strength necessary

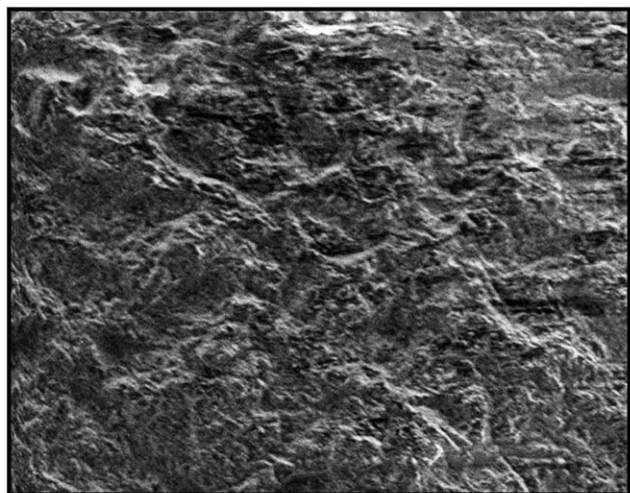


Figure 1. SEM micrograph (secondary electron mode at 750 \times) of resin composite surface treated with airborne particle abrasion with 50 μ m aluminum oxide particles.

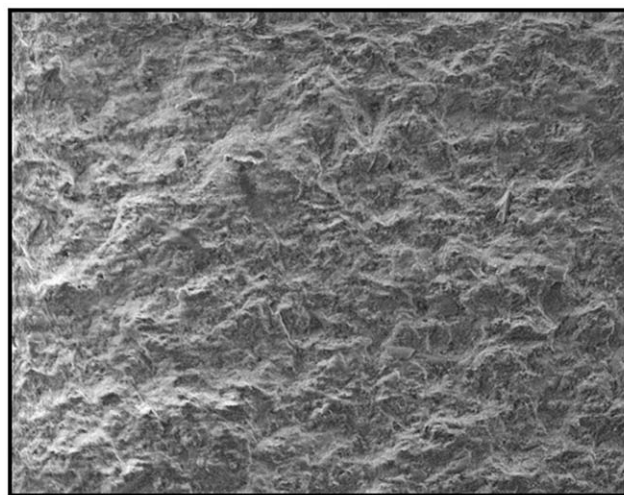


Figure 2. SEM micrograph (secondary electron mode at 750 \times) of resin composite surface treated with tribochemical silica coating.

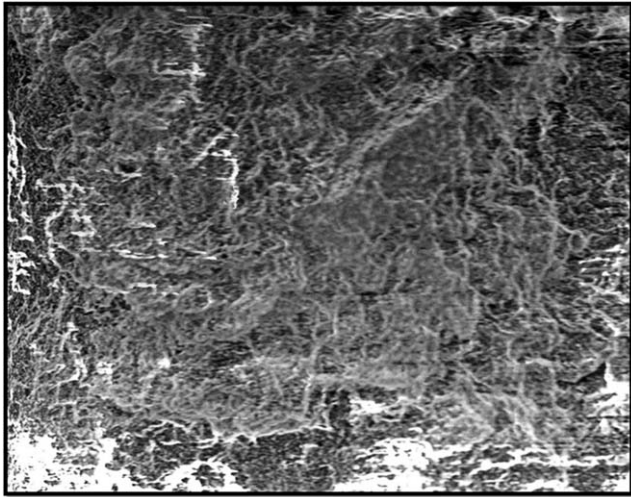


Figure 3. SEM micrograph (secondary electron mode at 750x) of resin composite surface treated with Er,Cr:YSGG laser.

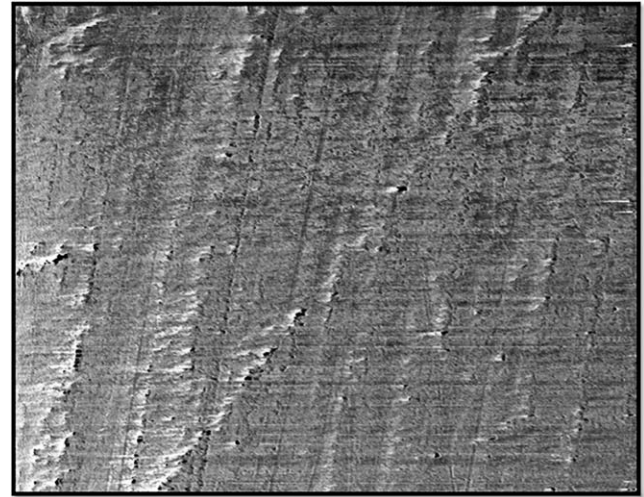


Figure 5. SEM micrograph (secondary electron mode at 750x) of resin composite surface without surface treatment.

for repairing resin composite restorations *in vivo* has not yet been defined, the surface treatment that produces the highest repair bond strength should be considered the most promising option.

Surface Treatment

In this study, air abrasion with 50- μ m aluminum oxide particles significantly improved the repair bond strength of the specimens when compared to the control. These results are in agreement with the majority of available studies.^{16–21} This improvement is believed to be due to the fact that air abrasion with aluminum oxide particles creates a roughened surface with microretentive areas. The SEM micrograph utilizing the secondary electron mode showed

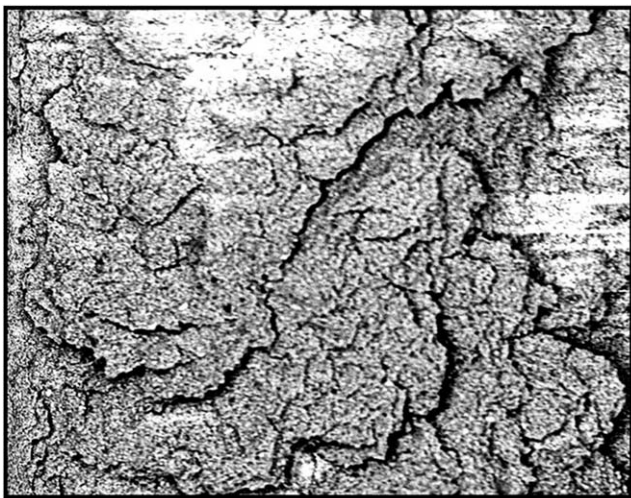


Figure 4. SEM micrograph (backscattered electron mode at 750x) of resin composite surface treated with Er,Cr:YSGG laser.

a generalized irregular surface (Figure 1) that could increase the wettability for an adhesive agent.

Tribochemical silica utilizes air abrasion with 30- μ m aluminum oxide particles modified with silica.²² This treatment increases the surface energy of aged composites and embeds a proprietary silicate ceramic layer of submicron particles which combine with silanization to provide a chemical and micromechanical bond for the repair of resin matrix composites. Some studies using the above agent show equivalent or superior results to air abrasion.^{23,24} However, Rathke and others²⁵ found that CoJet with silanization was not superior to bur or air abrasion when used with an adhesive agent. Moreover, if exposed enamel or dentin were involved, the use of silica coating and silanization would interfere with the resin matrix composite bond to the enamel or dentin at the repaired sites.

Surface treatment with tribochemical silica coating significantly improved the repaired bond strength when compared to the control group in the current study. This result is supported by other studies that found this technique effective.^{26,27} It was concluded that the effectiveness of this technique comes from the ability to create microirregularities, increase surface energy, and embed a proprietary silicate ceramic layer. The surface roughness value and the SEM characteristics of this surface treatment using the secondary electron mode revealed less irregular surface when compared with 50- μ m aluminum oxide particle air abrasion (Figure 2, Table 3). The present results also show that A50 had a slightly higher repaired shear bond strength than ATS, which agreed with the study by Rathke

Table 3: Mean Surface Roughness of Each Surface Treatment

Surface Treatments	Mean Surface Roughness/Ra value, μm
Air abrasion (A50)	2.27 ± 0.07
Tribochemical silica coating (ATS)	1.58 ± 0.03
Er,Cr:YSGG laser (ECL)	13.98 ± 0.95
No treatment (NST)	0.32 ± 0.10

and others.²⁵ They found that tribochemical silica coating was inferior due to the smaller particle size and shallower abrasion of 30- μm particles when compared with 50- μm particles. One might expect that tribochemical silica coating should be more effective when used with a silane coupling agent to promote a chemical interaction of the ceramic surface to a resin bonding agent; however, the application of silane to ATS did not show any significant difference in the present study.

Er,Cr:YSGG laser is another system with surface preparation potential. This type of laser (wavelength 2.78 μm) can be effectively absorbed by water and also hydroxyapatite crystal. The absorption of photon energy causes vaporization, resulting in macroscopic and microscopic irregularities via microexplosions of the material surface. The Er,Cr:YSGG pulsed laser, using an air-water spray, can cut enamel, dentin, cementum, bone, and soft tissue efficiently without significant thermal side effects. This laser operates in a pulse mode which achieves energy up to 300 mJ, with power up to 6 W.²⁸ Some studies have reported that tooth surfaces irradiated by Er,Cr:YSGG at a 90° angle showed a characteristic rough and clean surface without debris.^{29–31} In addition to soft and hard tissue surgeries, erbium laser can be used to remove resin matrix composite. The previous version of erbium, Er:YAG (erbium: yttrium-aluminum-garnet), was studied for effectiveness of resin matrix composite removal.^{32,33} Er,Cr:YSGG lasers are generally set at the following parameters: pulse duration 140 μs , a repetition rate of 20 Hz, a power output range from 0.0 to 6.0 W, pulse energy at 0 to 300 mJ per pulse and an energy density per pulse from 0.0 to 68.02 J/cm². Light delivery is through a fiberoptic system to a terminal sapphire tip with an adjustable air-water spray handpiece. Recent studies have set the power output

at 6.0 W with 90% air pressure and 75% water pressure for enamel etching,³⁴ and 4.0 W with 65% air pressure and 55% water pressure for dentin etching.^{35,36}

The results of the current study show no increase in repair shear bond strength when Er,Cr:YSGG laser was used. Air abrasion with 50- μm aluminum oxide particles and tribochemical silica resulted in significantly higher repair shear bond strength than both Er,Cr:YSGG laser and control. There was no significant difference in shear bond strength between the Er,Cr:YSGG laser group and the control group.

The surface roughness value of the laser irradiated specimens revealed the highest Ra values (Table 3), while the SEM micrograph (secondary electron mode) showed surfaces with the most irregular surface. Interestingly, cracks were only observed in the SEM micrographs (backscattered electron mode) with the Er,Cr:YSGG laser treatment, but not with other surface treatments (Figure 4).

Silanization

A silane coupling agent is a molecule with two functional groups, a silanol group for reacting with inorganic filler particles, and an organofunctional group for reacting with the methacrylate in the resin matrix. Silane has been used clinically for promoting a covalent bond between porcelain and resin for ceramic repair. It has been shown to improve bond strength between resin matrix composites after bur abrasion^{37,38}, airborne particle abrasion³⁹ and after hydrofluoric acid application due to the exposure of filler particles. Silanization in the current study did not significantly improve repair bond strength for any groups. This result agreed with the previous study by Brosh and others⁴⁰ that using a silane coupling agent showed no significant improvement of bond strength. A prior study by Swift and others⁴¹ found lower repaired bond strengths when using a silane coupling agent after air abrasion. Bonstein and others⁴² proposed that silane might change the structure of the matrix of aged resin composite and leave the filler particles bare, leading to a reduction in retention.

Based on these studies, it can be theorized that the cohesive failure in the control groups might be caused by two factors. Firstly, insufficient aging from the thermocycling process occurred. The thermocycling took approximately 52 hours for 5000 cycles, which might have been enough to create thermal stress to the resin composite, but may not

have impacted all unreacted carbon-carbon double bonds. Secondly, the bonding agent (OptiBond Solo Plus, Kerr) might be very effective in penetrating into microcracks on the surface and creating a strong covalent bond to aged resin composite.

CONCLUSIONS

- 1) The use of airborne particle abrasion and tribochemical silica coating in combination with a bonding agent significantly increased the repair bond strength.
- 2) Surface treatment with Er,Cr:YSGG did not show any improvement in bond strength.
- 3) Silanization did not improve the repair bond strength.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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