Microtensile bond strength of repaired indirect resin composite

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PURPOSE. The objective of this study was to investigate the effect of surface treatments on microtensile bond strengths (MTBSs) of two types of indirect resin composites bonded to a conventional direct resin composite. **MATERIALS AND METHODS.** Indirect resin composite blocks of Ceramage and SR Nexco were prepared in a plastic mold having a dimension of 10 × 10 × 4 mm. These composite blocks were divided into three groups according to their surface treatments: Group1: Sandblast (SB); Group2: Sandblast and ultrasonically clean (SB+UL); Group3: Sandblast plus silane (SB+SI). After bonding with direct resin composite, indirect-direct resin composite blocks were kept in distilled water for 24 hours at 37°C and cut into microbars with the dimension of 1 × 1 × 8 mm. Microbar specimens (n = 40 per group) were loaded using a universal testing machine. Failure modes and compositions were evaluated by SEM. The statistical analyses of MTBS were performed by two-way ANOVA and Dunnett's test at α = .05. **RESULTS.** Surface treatments and brands had effects on the MTBS without an interaction between these two factors. For SR Nexco, the MTBSs of SB and SB+SI group were significantly higher than that of SB+UL. For Ceramage, the MTBSs of SB and SB+SI were significantly higher than that of SB+UL. The mean MTBS of the Ceramage specimens was significantly higher than that of SR Nexco for all surface treatments. **CONCLUSION.** Sandblasting with or without silane application could improve the bond strengths of repaired indirect resin composites to a conventional direct resin composite. ***[J Adv Prosthodont 2017;9:38-44]***

KEYWORDS: Surface treatment; Indirect resin composite; Microtensile bond strength; Sandblast; Silane; Ultrasonic

INTRODUCTION

Indirect resin composites (IRCs) are used to fabricate many kinds of both intracoronal and extracoronal dental restorations including inlays, onlays, overlays, veneering material for fixed restorations, and removable dentures. During 1960s and 1990s, the first and the second generations of indirect resin composites were developed to improve both their physical and mechanical properties. For the second generation of indirect resin composites, an increase in micro-hybrid fillers and a decrease in organic matrix were aimed to improve the mechanical strength and to decrease polymerization shrinkage of these materials.[1,2]

Dental restorations made from indirect resin composite offer some benefits as compared to direct resin composite restorations, such as better mechanical performance and a significant reduction in polymerization shrinkage. Therefore, they could provide longer service time and better color stability and would reduce postoperative sensitivity.[3-5] It is also easier to achieve ideal proximal contacts and anatomic morphology, precise marginal integrity, and optimal esthetics.[6]

Compared to ceramic materials, indirect resin composites exhibit better stress distribution, better reparability, lower cost, and ease of handling.[7,8] Due to their low elastic moduli, resin composite materials have shown a greater capacity to absorb compressive loading forces and reduce the impact forces than porcelain has. Due to similar composition of the luting cement and composites, the marginal adaptation of composites is better than that of ceramics and resin composites have a lower tendency for marginal chipping than ceramics.[4,6] On the other

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hand, resin composites show inferior long term surface characteristics, such as surface roughness and esthetics, and they are more prone to color changes.[1,9]

In recent years, indirect resin composites have gained more popularity because of their improved wear resistance similar to natural tooth and also because they can overcome the problem of veneering porcelain, including tendency to wear opposing teeth significantly and difficulty of repair.[10] Despite the considerable improvement of indirect resin composites, clinical failures may still occur, including bulk fractures, chipping, marginal gaps, wear, and color alteration. In such situations, it may be necessary to replace or repair the existing restorations in order to restore function and esthetics. Successful resin composite repairing procedure requires an adequate interfacial bond between existing and new resin composite materials. Various methods have been reported to improve the reactivity of highly converted composites. These methods include acid etching, air abrasion, and the use of solvents and silanes.[10-13] There is no consensus on the results obtained from these procedures.

Hydrofluoric acid has capacity of increasing surface roughness in the aged resin composite surface by the dissolution of the filler particles. Many studies have shown that sandblasting and silanization can promote durable bond strength of repaired composite without HF etching, and thus using hazardous and highly corrosive HF can be avoided.[11-13] Hummel *et al.*,[14] in 1997, reported that surface treatment by hydrofluoric acid or phosphoric acid etching alone was not sufficient to create an effective bond for repaired indirect composite. In addition, Cesar *et al.*,[11] in 2001, studied the tensile bond strength of composite repairs applied to Artglass, and they reported that using silane after sandblasting resulted in statistically higher bond strength compared to using sand paper, diamond bur, and acid etching surface treatment.

Although some studies mentioned that none of the surface treatment could be recommended as a universal protocol for repairing resin composites, knowing compositions of repaired composites would be helpful for repairing procedure.[15] The studies suggested that when the composition of a repaired composite was unknown, etching with phosphoric acid or sandblasting was recommended.[15] In addition, silica coating, or aluminum oxide blasting and silanization provided significantly better results in repaired indirect resin composite.[10-11,13,16-17]

SR Nexco and Ceramage are the new materials in the family of the second generation indirect resin composites. The higher amount of ceramic fillers and the use of UDMA resin monomers are the main improvement of these indirect resin composites. Because of their improved mechanical properties, it is claimed that they can be used to fabricate various kinds of dental restorations. However, there is limited information regarding their clinical performance. The objectives of this study were to compare the microtensile bond strengths of two new indirect resin composite (Ceramage and SR Nexco) repaired with direct resin composite (Filtek Z350). Their failure characteristics were also determined.

MATERIALS AND METHODS

Indirect resin composite blocks of Ceramage (Lot No. 081/23, Shofu Inc., Kyoto, Japan) and SR Nexco (Lot No. R31493, Ivoclar Vivadent, Schaan, Liechtenstein) were prepared in a plastic mold (10 mm × 10 mm × 4 mm). The compositions of Ceramage and SR Nexco are shown in Table 1. Incremental layers (2 mm) of indirect resin composite were condensed into a mold using a load transfer device at the force of 1 kg. Then, each layer was initially polymerized using a hand-held light curing device for 40 s (Optilux 501, Kerr, Danbury, CT, USA). The last increment was covered with a glass slab in order to obtain a flat surface after light curing. The intensity of the light was verified to be no less than 500 mW/cm^2 by using a built-in radiometer before starting the polymerization procedure. After a specimen was removed from the mold, Ceramage oxy-barrier (Lot No. R60504, IvoclarVivadent AG, Schaan, Liechtenstein) or SR gel (Lot No. R60504, Ivoclar Vivadent AG, Schaan, Liechtenstein) was applied to prevent oxygen inhibited layer and the specimen was further polymerized in a special polymerization device (Solidilite V, Shofu Inc., Kyoto, Japan) following the manufacturer's recommendations. After complete polymerization, the outer surfaces were polished with silicone carbide papers 600 grit under running water. Then all specimen blocks were stored in distilled water for 24 hours at 37°C in an incubator.

These blocks were categorized into three groups according to their surface treatments (2 blocks per group).

Group 1: Sandblast (SB):

Specimens were sandblasted with aluminum oxide particles (Al_2O_3) (Tecline, Bandhagen, Sweden) of 50 μm, 60 psi at a distance 1 cm perpendicular to surface for 5 seconds by a sandblasting machine (Base M.B.L, Dentalfarm, Torino, Italy)

Table 1. Compositions of indirect resin composite use in this study

Material	Manufacturer	Polymer matrix	Filler	Particle
SR Nexco Paste dentine	IvoclarVivadent, Schaan, Leichtenstein	UDMA, Aliphatic Dimethacrylate (16.9%wt)	silicon dioxide (19.8%wt) prepolymer and co-polymer which consists of pre-polymerised ground up UDMA matrix and inorganic microfiller particles (62.9%wt.)	10 - 50 nm
Ceramage	SHOFU Inc., Kyoto, Japan	UDMA	zirconium silicate (73%wt)	NP

NP = not provided by the manufacturers

Group 2: Sandblast and ultrasonic clean (SB+ UL):

After sandblasted, composite specimens were cleaned ultrasonically in distilled water for 10 minutes using an ultrasonic cleaner (Vibraclean 300, MDT Co., Harvey, CA, USA) and steam-cleaned for 5 seconds.

Group 3: Sandblast plus silane (SB+SI):

After sandblasted, composite specimens were applied with silane (Monobond-S, Ivoclar Vivadent, Germany) using a microbrush, left for 60 seconds, and dispersed with a strong air stream.

After surface treatments, indirect resin composite blocks were positioned in another plastic mold (10 mm × 10 mm × 9 mm). The direct resin composite (Filtek Z350, Lot No. 441110, 3M ESPE, St. Paul, MN, USA) was condensed on the treated surface of indirect resin composite by incremental technique without bonding agents. Each layer was light-polymerized for 40 seconds (Optilux 501, Kerr, Danbury, CT, USA) until completion. The last increment was covered with a glass slab in order to obtain a flat surface. After the specimens were removed from the mold, they were further polymerized for 40 seconds in the areas that previously contacted with the plastic mold.

The specimens were stored in distilled water for 24 hour at 37°C in incubator and cut into microbars of 8 mm length and 1 mm^2 in cross-section with the Micro Cutting Instrument (Struers, Copenhagen, Denmark). Sizes of all microbars were measured with a digital caliper and were recorded. Only microbars with cross-sectional area between 1.0 ± 0.1 mm^2 were selected for MTBS test. All microbars were examined for defects with an optical light microscope at 10× magnification. The microbars with any defects were discarded from the study. The number of microbars tested in each group was forty.

The microtensile bond strength (MTBS) test was performed using a universal testing machine (Model 5566, Instron Ltd., Buckinghamshire, England) in tension at a crosshead speed of 1 mm/min. The maximum load at failure was recorded and the tensile bond strength was calculated using the formula:

$R = F/A$

Where "R" is the MTBS (in MPa), "F" is the load at fracture (in N) and "A" is the interfacial area of the specimen (in mm^2)

The scanning electron microscope (SEM) was used to evaluate compositions of indirect resin composites. Modes of failure were identified using SEM. The failure modes were classified into three modes:

Cohesive failure of indirect resin composite: where fracture occurred in indirect resin composite

Cohesive failure of direct resin composite: where fracture occurred in direct resin composite

Interfacial failure: where fracture occurred at the interface between indirect and direct resin composite

The mean MTBSs and standard deviations were calculated and recorded. Shapiro-Wilk test was used to test the normality of the data and Levene's test was used for testing the equality of variances. The MTBS values of all groups were analyzed by two-way ANOVA and Dunnett's T3 multiple comparison test at $\alpha = .05$

RESULTS

The mean MTBSs of all groups are shown in Table 2. The results from two-way ANOVA showed that different brands of indirect resin composite (SR Nexco and Ceramage) and surface treatments (SB, SB+UL and SB+SI) had effects on the MTBS without an interaction between these two factors. The MTBS of Ceramage was higher than SR Nexco ($P < .001$) for all surface treatments. Dunnett's T3 multiple comparison test was used to identify the difference between MTBSs of different surface treatment groups and the results showed that the mean MTBSs of SB and SB+SI groups were significantly higher than that of SB+UL group ($P < .001$) for both SR Nexco and Ceramage. The failure modes of fracture specimens are presented in Table 3 and Table 4. The number and percentage of specimens according to their failure mode are shown in Table 3. Cohesive failures of direct resin composite and interfacial failures were mostly observed for Ceramage material. For SR Nexco, cohesive failure of indirect resin composite and interfacial failure were frequently observed. The mean MTBS values of specimens in all groups according to their failure mode were calculated as shown in Table 4. Slight variation in the mean MTBS values for each material was observed depending on the failure mode. The microstructures of indirect and direct resin composites are shown in Fig. 1, Fig. 2, Fig. 3, Fig. 4. The irregular shape SiO_2 particles with varied particle sizes, ranging from submicron size to nearly 50 μm were observed in SR Nexco (Fig. 1, Fig. 3). In Ceramage, the round-shape zirconium silicate particles less than 10 μm in size are observed. (Fig. 2, Fig. 4) The SEM micrographs of direct resin composite (Filtex Z350) showed irregular shape of zirconium silicate particles, mostly less than 10 μm in size (Fig. 1).

Table 2. Mean microtensile bond strengths (in MPa) (standard deviations in parentheses) of six experiment groups

Indirect resin composite	Surface treatments		
	SB	SB + UL	SB + SI
SR Nexco	53.04 (10.52)[a]	43.81 (10.83)[b]	53.23 (10.45)[a]
Ceramage	72.08 (14.52)[c]	63.88 (12.90)[d]	72.49 (15.70)[c]

Groups with different superscript letters indicate significant differences ($P < .05$).

SB = Sandblast only, SB + UL = Sandblast and ultrasonic clean application, SB + SI = Sandblast plus silane application

Table 3. The number and percentage of failure mode of six experiment groups

Indirect resin composite	Surface treatments	Failure mode		
		Interfacial	Cohesive of direct resin composite	Cohesive of indirect resin composite
SR Nexco	SB	11 (23.40 %)	8 (17.02 %)	28 (59.57 %)
	SB + UL	26 (54.16 %)	9 (18.75 %)	13 (27.08 %)
	SB + SI	11 (25 %)	8 (18.18 %)	25 (56.82 %)
Ceramage	SB	25 (52.08 %)	14 (29.17 %)	9 (18.75 %)
	SB + UL	15 (33.33 %)	24 (53.33 %)	6 (13.33 %)
	SB + SI	9 (21.43 %)	23 (54.76 %)	10 (23.81 %)

Table 4. The mean (standard deviation in parentheses) of MTBS according to the failure mode

Indirect resin composite	Surface treatments	Failure mode		
		Interfacial (Mixed)	Cohesive of direct resin composite	Cohesive of indirect resin composite
SR Nexco	SB	53.92 (10.91)	54.21 (9.63)	52.36 (10.91)
	SB + UL	44.10 (13.63)	41.17 (7.72)	45.14 (6.07)
	SB + SI	56.06 (9.94)	50.89 (11.50)	52.74 (10.50)
Ceramage	SB	74.65 (16.64)	70.58 (10.12)	67.27 (13.83)
	SB + UL	68.94 (14.78)	60.88 (9.28)	63.23 (18.35)
	SB + SI	73.97 (17.75)	70.88 (13.92)	74.85 (18.80)

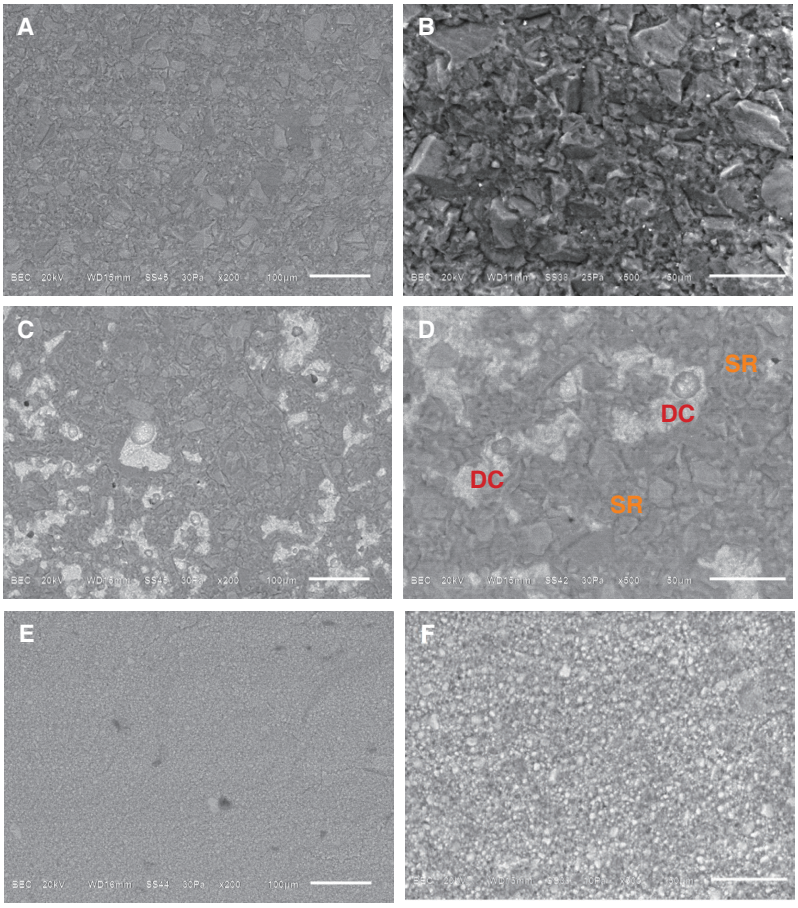

Fig. 1. The representative SEM photographs of SR Nexco groups. (A), (B) the cohesive failure in SR Nexco (SR). (C), (D) the interfacial failure. (E), (F) the cohesive failure in the direct resin composite (DC).

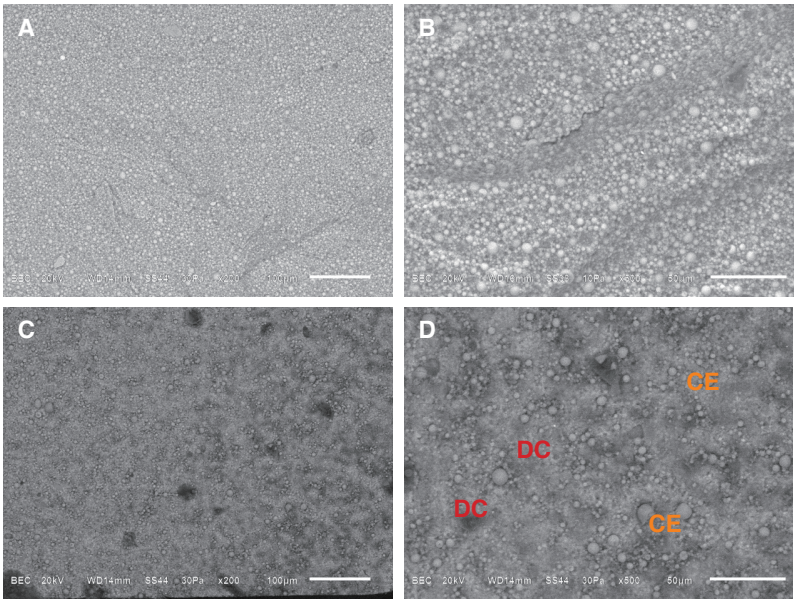

Fig. 2. The representative SEM micrographs of Ceramage groups. A, B: the cohesive failure in Ceramage (CE). C, D: the interfacial failure.

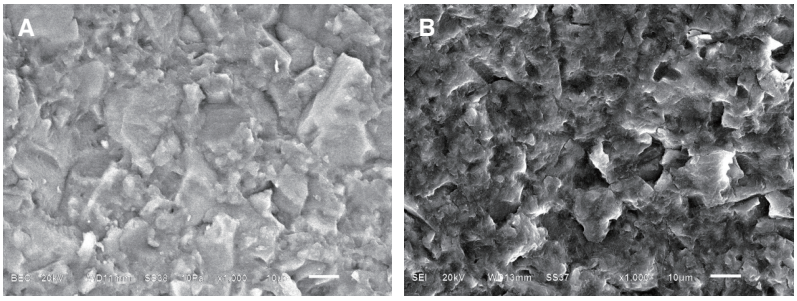

Fig. 3. The representative SEM micrographs show microstructures of SR Nexco in backscattered electron composition (BEC) and secondary electron imaging (SEI) mode.

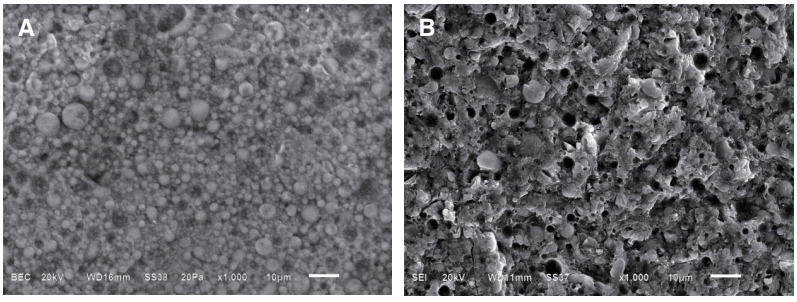

Fig. 4. The representative SEM micrographs show microstructures of Ceramage in backscattered electron composition (BEC) and secondary electron imaging (SEI) mode.

DISCUSSION

Several techniques are purposed to improve the repair bond strength of indirect resin composites including mechanical interlocking and chemical bonding of resin composite materials.[10] However, the success of composite-composite adhesion depends on the surface treatment of an aged resin composite.[18] The bonding between aged and new resin composite could be accomplished by three mechanisms, which are a chemical bonding with the organic matrix, a chemical bonding with the exposed filler particles, and the micromechanical retention to the treated surface.[19] A chemical bonding with the organic matrix relies on the unconverted C=C double bonds remaining in the surface of the aged composite.[18] The higher conversion rate of an indirect resin composite may compromise its repairing procedure.[10]

For a chemical bonding with the exposed filler particles, silane is effective in improving adhesion to silica-based materials. Having the structure Y-Si(OR),[3] where Y is a functional group (usually be methylmethacrylate), silane will chemically react with the adhesive resin (dimethacrylate). OR is an alkoxyl group, which is hydrolyzed to a silanol (SiOH) and forms siloxane bonds with silanols on the filler particles surface. Therefore, it is possible to create chemical bond with the filler particles of the aged composite using a silane coupling agent, and silane also improves the wettability of the substrate surfaces.[12]

For micromechanical retention to the treated surface, surface conditioning, such as the use of diamond burs, sandblasting, and acid etching, can create micromechanical retention and increase the bond strength of repaired composite. Few studies have shown that the surface grinding of the indirect resin composite with diamond bur would yield less bond strength than the others.[11]

In this study, pre-testing failures were found in the untreated indirect-direct resin composite specimens, as well as in the resin treated with silane only. Bonding failures in the untreated indirect-direct resin composite specimens were found during the polishing procedure with a silicon carbide paper. The specimens treated with silane only had bonding failures during the cutting process. Therefore, no treatment or surface treatment by silane alone could not be included in this study because all the specimens were debonded during specimen preparation steps. Along with previous studies, using silica coating or aluminum oxide blasting increased microtensile bond strength values of repaired direct resin composite, irrespective of primer used.[6,11,20-21] Alumina particles have been used to increase surface roughness of the substrates and some alumina particles might have been embedded into the surfaces during grit blasting and formed =Al-O-Si bonds after silanization.[10,12] Thus, sandblasting was a necessary process for bonding of indirect-direct resin composite.[13]

According to the manufacturers' information, flexural strength of Ceramage (146 MPa) is higher than that of SR Nexco (90 MPa). The main reason for the differences in strength should be the differences in compositions of these materials; SR Nexco Paste has highly dispersed silicon dioxide as microfiller in the 10 to 50 nm range. The main filler component (62.9%) is a prepolymer/copolymer, which consists of pre-polymerized ground up UDMA matrix and inorganic microfiller particles. On the other hand, Ceramage is composed of zirconium silicate particles supported by an inorganic polymer matrix (UDMA). The microstructures of indirect and direct resin composites are shown in Fig.1, Fig. 2, Fig. 3, Fig. 4. SiO2 particles of SR Nexco are varied in size and non-homogeneous because the prepolymer and copolymer are present in the composition of the resin. For Ceramage material, zirconium silicate particles with sizes ranging between submicron to approximately 10 μm were observed.

The significantly higher bond strengths of repaired indirect resin composite were observed when SR Nexco and Ceramage were treated with sandblast only or sandblast plus silane application. This result was in accordance with previous studies that showed the higher bond strength when using sandblasting and silane as opposed to hydrofluoric acid, diamond bur, or other procedures to create micromechanical retention.[11,13,21] However, this result showed that silane would not be necessary for repairing resin composite as also reported in previous studies.[16,22] Surface treatment by alumina sandblasting alone is sufficient to produce effective bond strength for repairing indirect resin composite with direct composite.

From this study, sandblasting and ultrasonic clean had the lowest microtensile bond strength in both Ceramage and SR Nexco. Moisture at the interface from ultrasonic cleaning of repaired indirect resin composite might be the reason for reduced bond strength. Water absorption affects the bond strength by hydrolytic degradation of the resin-filler interface and causes swelling of the resin matrix that could lower the bonding ability and mechanical properties of resin composite materials.[23-25] However, actual ultrasonic cleaning cannot be used in the patients with the direct repairing technique but can be used only in a laboratory.

The failure modes of Ceramage were mostly cohesive failure of direct resin composite or interfacial failure. On the other hand, failure modes of SR Nexco were mostly cohesive failure of indirect resin composite or interfacial failure. Considering the tensile strength values of all materials used in this study, direct resin composite and Ceremage were the strongest with tensile strength approximately 62 - 80 MPa, according to the manufacturers' information. SR Nexco was the weakest material. The factor that convincingly determined the bond strength of indirect-direct resin composite bonding would be the strength of these resin composite materials. When SR Nexco was bonded to direct resin composite, failure usually occurred in SR Nexco, which was the weaker material. When Ceramage was bonded to direct resin composite, failure could occur in both materials because they had comparable strength. Therefore, mechanical properties of resin composite materials would be an important factor that control the successive bonding of these materials.

CONCLUSION

Surface treatment methods and compositions of indirect resin composite had effects on the MTBS of bonded indirect-direct

resin composite bilayers.

Sandblasting with or without silane application could improve the bond strengths of repaired indirect–direct resin composites in both SR Nexco and Ceramage.

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