



Effect of surface treatments on shear bond strength of resin composite bonded to CAD/CAM resin-ceramic hybrid materials

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PURPOSE. The purpose of this study was to assess the effect of surface treatments on shear bond strength of resin composite bonded to thermocycled and non-thermocycled CAD/CAM resin-ceramic hybrid materials.

MATERIALS AND METHODS. 120 specimens (10×10×2 mm) from each material were divided into 12 groups according to different surface treatments in combination with thermal aging procedures. Surface treatment methods were airborne-particle abrasion (abraded with 50 micron alumina particles), dry grinding (grinded with 125 µm grain size bur), and hydrofluoric acid (9%) and silane application. According to the thermocycling procedure, the groups were assigned as non-thermocycled, thermocycled after packing composites, and thermocycled before packing composites. The average surface roughness of the non-thermocycled specimens were measured after surface treatments. After packing composites and thermocycling procedures, shear bond strength (SBS) of the specimens were tested. The results of surface roughness were statistically analyzed by 2-way Analysis of Variance (ANOVA), and SBS results were statistically analyzed by 3-way ANOVA. **RESULTS.** Surface roughness of GC were significantly lower than that of LU and VE ($P<.05$). The highest surface roughness was observed for dry grinding group, followed by airborne particle abraded group ($P<.05$). Comparing the materials within the same surface treatment method revealed that untreated surfaces generally showed lower SBS values. The values of untreated LU specimens showed significantly different SBS values compared to those of other surface treatment groups ($P<.05$). **CONCLUSION.** SBS was affected by surface treatments. Thermocycling did not have any effect on the SBS of the materials except acid and silane applied GC specimens, which were subjected to thermocycling before packing of the composite resin. [J Adv Prosthodont 2016;8:259-66]

KEYWORDS: Surface treatment; Shear bond strength; Resin ceramic hybrids

INTRODUCTION

The esthetic expectation resulted in an increase in metal-free tooth colored materials.¹ Although all-ceramic restorations generally meet these esthetic expectations, a major drawback is their brittleness, which resulted from the low tensile stress

and fracture toughness of the material.² Currently, ceramic restorations are frequently milled out of industrially made computer aided designed and manufactured ceramic blocks. Processing ceramics under industrial conditions improves mechanical properties in comparison with laboratory process.^{3,4} Besides different type of ceramic blocks (feldspathic ceramic, reinforced glass ceramics, zirconia, etc.), new materials, namely resin-ceramic hybrid materials, have been developed for CAD/CAM (computer aided design and computer aided manufacturing) technique. These materials combine the advantageous properties of ceramics, such as durability and color stability, with those of composite resins, such as improved flexural properties and low abrasiveness.^{5,6} Vita Enamic (Vita Zahnfabrik, Bad Sackingen, Germany), Lava Ultimate (3M-ESPE, Seefeld, Germany), and GC Cerasmart (GC Dental Products, Leuven, Belgium) blocks are recent CAD/CAM materials that provide superior esthetic appearance.⁷⁻⁹ According to the manufacturer's

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information, Vita Enamic (VE) is a resin infused ceramic, and its composition is approximately 14% composite distributed into a ceramic network (86 wt%).¹⁰ Lava Ultimate (LU) blocks are composed of nanoceramic particles (80%) embedded in a highly cured resin matrix (20%), and GC Ceresmart (GC) is a high-density composite resin material containing 71% filler particles by weight.^{8,9}

In the case of all commercially available ceramics, CAD/CAM restorations in clinical service are also susceptible to fractures because of trauma, parafunctional habits, etc. Because of the nature of the ceramic processing, new porcelain cannot be added to an existing restoration intraorally. Thus, intraoral repair can be considered as an emergency treatment for localized fractures.^{11,12} Furthermore, it represents a viable alternative to remake of a fractured all-ceramic restoration because the removal of the restoration is difficult and replacement is expensive for these restorations.¹²⁻¹⁷ Surface treatment on the fractured ceramic surface must be performed in the repair procedure. It involves mechanical or chemical treatments to create irregularities on the surface. Bonding components are also required for the adhesion to a restorative material.¹⁸ Roughening with diamond burs,¹⁹ etching with hydrofluoric acid,^{20,21} and sandblasting²¹ are the surface treatments recommended for ceramics. Silane is also used for ceramics in order to strengthen the bond between the ceramic and cement. It can increase the surface energy for adhesive application.²² Although the intraoral repair process for ceramic restorations has been widely investigated in the literature,^{18,21,23-26} limited information is available on the surface treatments and intraoral repair protocol for CAD/CAM resin-ceramic hybrid materials.^{13,27} The aim of this *in-vitro* study is to evaluate the effect of surface treatments on SBS of resin composite bonded to thermocycled and non-thermocycled CAD/CAM resin-ceramic hybrid materials and to determine the effect of surface treatments on the surface roughness of these hybrid ceramics.

The null hypotheses of this study were that (1) type of material, (2) type of surface treatment, and (3) aging would not affect the SBS of a composite resin to CAD/CAM resin-ceramic hybrid materials.

MATERIALS AND METHODS

Three CAD/CAM resin-ceramic hybrid materials (LU, VE, and GC) were selected for the study. From each material, 120 specimens (10 × 10 × 2 mm) were prepared from pre-fabricated blocks using a low speed cutting device (Microcut Precision Cutter, Metkon, Bursa, Turkey) under water cooling. Specimens were embedded in acrylic resin (Panacryl, Arma Dental, İstanbul, Turkey) blocks and then polished with #600 SiC polishing paper in cold flowing water. The 120 specimens of each material were divided into 12 subgroups (n = 10) according to different surface treatments in combination with thermal aging procedures. Grouping of specimens is schematically shown in Figure 1.

The specimens in each thermal aging group (thermocyc-

led before application of repair composite resin or thermocycled after application of repair composite resin) were thermocycled between 5°C and 55°C for 10000 cycles with a 20 seconds dwell time in a thermocycler (MTE 101; MOD Dental, Esetron Smart Robotechnologies, Ankara, Turkey).

Materials in non-thermocycled, thermocycled before application of composite resin, and thermocycled after application of composite resin groups were assigned to 4 subgroups according to surface treatment method:

- (1) No treatment
- (2) Airborne-particle abrasion using 50 micron Al₂O₃ particles (Korox 50; BEGO, Bremen, Germany) at 4 bar pressure for 10 seconds from a distance of 10 mm perpendicular to the surface of the specimen
- (3) Dry grinding with green banded (125 µm grain sized) diamond bur by taking away a very thin layer from the surface
- (4) Hydrofluoric acid and silane application. The surfaces were conditioned with 9% hydrofluoric acid (Ultradent Porcelain Etch, South Jordan, UT, USA) for 60 seconds and subsequent silanization (Ultradent Silane; South Jordan, UT, USA) was performed for 60 seconds.

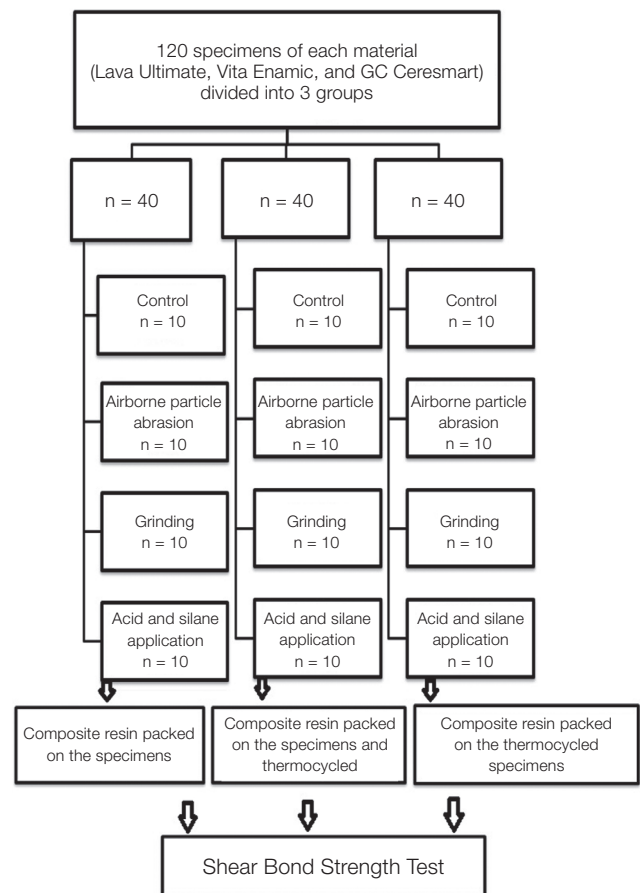


Fig. 1. Schematic diagram of experimental groups.

After surface treatments, all specimens were cleaned with ethanol and an ultrasonic cleaner (Erosonic Energy; Euronda, Vicenza, Italy) in distilled water for 10 minutes. A composite resin (Clearfil Majesty Esthetic; Kuraray, Medical, Tokyo, Japan) cylinder was packed onto specimen surfaces using a cylindrical metal mold that was 6 mm in diameter and 2 mm in depth. The composite resin layers were incrementally condensed into the mold, and each layer (1 mm) was cured for 20 seconds using a light curing unit (DB-686latte; Foshan Coxo Medical Instrument Co. Ltd., Foshan City, China). After curing, the CAD/CAM resin-ceramic hybrid-composite resin block was removed from the mold (Fig. 2). Subsequently, all specimens were kept in 37°C distilled water for 24 hours to allow for post-polymerization.²⁷

The average surface roughness (in Ra) of the specimens not subjected to thermocycling was measured with a profilometer (MahrSurf M 300 C, Mahr GmbH, Göttingen, Germany) before application of repair composite resin. The probe was placed in the middle of the specimen surface, and measurements were made in different directions with a traversing length of 1.75 mm and a constant measuring speed of 0.2 mm/second.

The SBS was determined with a universal testing machine (Lloyd LF Plus; Ametek Inc., Lloyd Instruments, Leicester, England). The specimens were fixed by using a jig, and the interface between the specimens and resin was loaded at a crosshead speed of 0.5 mm/min. The maximum load (P) was measured when the composite resin cylinder was separated from the specimen surface.

The SBS was calculated from the following formula:

$$\text{SBS (in MPa)} = P \times 9.8 / r^2 \times \pi$$

where P is the maximum load (in kgF) and r is the radius (in mm) of the composite resin cylinder.

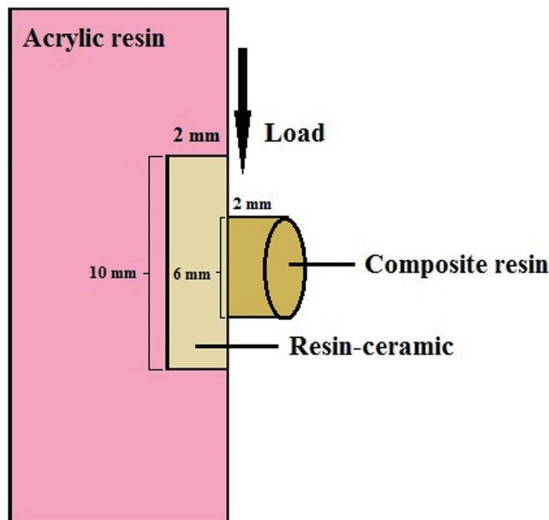


Fig. 2. Schematic diagram of shear bond strength test.

Surface roughness data were analyzed by 2-way ANOVA. SBS results were statistically analyzed by 3-way ANOVA with the material types, surface treatments, and thermocycling as the independent variables. Tukey test was used to determine any significant differences among the groups. All statistical analyses were carried out with a special software (SPSS 18.0 for Windows; IBM Corp., SPSS Inc., Chicago, IL, USA) at a significance level of $\alpha = .05$.

RESULTS

The results of the 2 way ANOVA showed that there was no interaction between the surface treatment methods and the materials ($P = .07$). Surface roughness values of surface treatments and materials are presented in Table 1 and Table 2. Among the surface treatments, the highest surface roughness was observed for dry grinding group, followed by airborne particle abraded group ($P < .05$). The lowest surface roughness were found in untreated (control) and acid and silane applied groups, and the value difference between the two groups was not significant ($P > .05$). When the materials were compared regardless of the surface treatment method, the difference was not significant between LU and VE, but GC showed significantly lower roughness than other materials ($P < .05$).

Table 1. Comparison of the average surface roughness (in Ra; μm) of the surface treatments regardless of the material type

Surface treatment (n = 30)	Mean (\pm SE)
Control	0.27 (0.03) ^C
Air-particle abrasion	1.52 (0.16) ^B
Grinding	4.15 (0.20) ^A
Acid and silane application	0.39 (0.05) ^C

SE: Standard Error of the mean

Same capital letters vertically indicate that average surface roughnesses (in Ra) were not significantly different among the surface treatments ($P > .05$).

Table 2. Comparison of the average surface roughness (in Ra; μm) of the materials regardless of the surface treatment methods

Material (n = 40)	Mean (\pm SE)
Lava Ultimate	1.76 (0.30) ^A
Vita Enamic	1.65 (0.26) ^A
GC cerasmart	1.34 (0.25) ^B

SE: Standard Error of the mean

Same small letters vertically indicate that average surface roughnesses (in Ra) were not significantly different among the materials ($P > .05$).

4 composites in LU control group and 1 composite in LU acid and silane applied group were failed during thermocycling. Furthermore, 1 composite in GC air-particle abraded group and 2 composites in LU control group were failed before loading of shear bond strength test. The failed specimens were excluded from the statistical analysis. Three-way ANOVA showed that there was an interaction between material, surface treatment, and thermocycling ($P < .05$) (Table 3).

The comparison of the shear bond strength values of the experimental groups are presented in Table 4.

The shear bond strength value of untreated LU group was significantly lower than the other materials ($P < .05$). The shear bond strengths of the airborne particle abraded groups were significantly different among the materials ($P < .05$). VE showed the highest shear bond strength values in grinding and acid and silane application groups while the results were not significantly different in no thermocycled-

Table 3. Values of the three-way ANOVA test for shear bond strength

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Material	2	984.31	1168.59	584.29	38.28	.000
Surface treatment	3	2565.91	2723.03	907.68	59.47	.000
Thermocycling	2	38.45	42.26	21.13	1.38	.252
Material * Surface treatment	6	1063.36	1016.79	169.46	11.10	.000
Material * Thermocycling	4	26.62	28.78	7.19	0.47	.757
Surface treatment * Thermocycling	6	77.47	76.54	12.76	0.84	.543
Material * Surface treatment * Thermocycling	12	537.89	537.89	44.82	2.94	.001
Error	316	4823.11	4823.11	15.26		
Total	351	101117.13				

Table 4. Comparison of the shear bond strength values among the materials, surface treatments, and aging groups

Surface Treatment	Control (non-thermocycled)			Thermocycling after application of composite resin			Thermocycling before application of composite resin		
	LU	VE	GC	LU	VE	GC	LU	VE	GC
	Mean (± SE)	Mean (± SE)	Mean (± SE)	Mean (± SE)	Mean (± SE)	Mean (± SE)	Mean (± SE)	Mean (± SE)	Mean (± SE)
Control	5.35 (0.53)	14.74 (1.13)	13.64 (1.24)	5.4 (0.85)	15.01 (1.22)	11.02 (1.04)	2.51 (0.37)	16.37 (1.04)	11.19 (1.2)
	(n = 9)	(n = 10)	(n = 10)	(n = 6)	(n = 10)	(n = 10)	(n = 9)	(n = 10)	(n = 10)
	B aY	A a Y	A a Y	B a Y	A a Y	A a Y	C a Y	A a XY	B a Z
Air-particle abrasion	15.59 (1.59)	16.8 (1.13)	16.08 (1.27)	15.79 (0.95)	13.95 (0.89)	15 (1.13)	15.13 (0.9)	14.78 (1.25)	15.96 (0.1)
	(n = 10)	(n = 10)	(n = 9)	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)
	A aX	A a Y	A a Y	A a X	A a Y	A a XY	A a X	A a Y	A a XY
Grinding	16.09 (1.14)	21.73 (0.91)	14.29 (1.21)	15.49 (1.22)	17.41 (1.41)	17.25 (1.54)	15.62 (1.28)	19.67 (1.25)	18.45 (1.68)
	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)
	B aX	A a X	B a Y	A a X	A a Y	A a X	A a X	A a X	A a X
Acid and silane application	16.28 (1.12)	17.91 (1.81)	20.64 (1.38)	16.34 (1.15)	22.16 (1.82)	16.31 (1.58)	16.63 (1.26)	19.73 (1.58)	13.64 (0.95)
	(n = 10)	(n = 10)	(n = 10)	(n = 9)	(n = 10)	(n = 10)	(n = 10)	(n = 10)	(n = 10)
	A aX	A a XY	A a X	B a X	A a X	B a X	B a X	A a X	B b YZ

SE: Standard of the mean.

Same capital letters horizontally indicate that shear bond strength values were not significantly different among materials in same surface treatment and aging group ($P > .05$).

Same small letters horizontally indicate that shear bond strength values were not significantly different among aging groups in same surface treatment and material ($P > .05$).

Same X, Y, Z letters vertically indicate that shear bond strength values were not significantly different among surface treatments in aging group and material ($P > .05$).

acid silane applied and both thermocycled-grinded groups ($P > .05$). Thermocycling decreased the shear bond strength values only in the acid and silane applied group in GC ($P < .05$). Surface treatments significantly increased the shear bond strength of LU ($P < .05$). Air particle abrasion did not significantly increase the shear bond strength of VE compared to control groups ($P > .05$). Grinding significantly increased the shear bond strength of VE in thermocycling group, but the difference was not significant in thermocycling groups. The highest shear bond strength values of GC were observed in acid silane applied groups in non-thermocycled group. The lowest values were observed in untreated and the highest values were observed in grinded specimens in thermocycling groups.

DISCUSSION

This *in vitro* study revealed that significantly different bond strength values were found when a composite resin was bonded to three CAD/CAM resin-ceramic hybrid materials. This study also demonstrated that surface treatments significantly increased the SBS of composite resin to CAD/CAM resin-ceramic hybrid materials compared to untreated material surface. These findings of this study led to rejection of the null hypotheses that there would be no difference on the shear bond strength based on type of material, type of surface treatment, and aging. This study was designed to investigate the intraoral reparability of recent CAD/CAM resin-ceramic hybrid materials because it is well known that intraoral repair is a minimally invasive and cost effective approach.¹⁵ The most crucial factor for evaluating the repair strength of restorative materials is mechanical interlocking.²⁷ Increasing surface roughness improves mechanical interlocking on the bonding surface.¹⁸ Therefore, different surface treatments were applied on the material surface and roughness of the treated surfaces was evaluated in the present study. Surface treatments, which include air-particle abrasion (50 μm Al_2O_3), grinding using green banded (125 μm grain sized) diamond bur, and acid etching (9% buffered hydrofluoric acid), were applied. These methods are commonly used surface treatments during intraoral repair of ceramic restorations.^{18,21} However, for CAD/CAM resin-ceramic hybrid materials, there is no agreement on the repair protocol that leads to favorable clinical outcome.^{7,13,27-29} These studies investigated an additional use of adhesive system to provide chemical bonding as well as mechanical pretreatment. The present study focused on bond strength of roughened surfaces, which is important in bonding procedure of restorative materials. Significantly highest roughness was found for grinded surfaces by diamond bur, followed by air-particle abraded surfaces. Untreated control and HF acid etched surfaces showed the lowest roughness values, the result which is similar to the previous studies.^{30,31} The literature includes limited information about effects of different surface treatments on the roughness CAD/CAM resin-ceramic hybrid materials. Frankenberger *et al.*⁷ recommended using hydrofluoric acid for Vita Enamic as well as

lithium disilicate ceramics and sandblasting for Lava Ultimate. This recommendation can be supported by surface roughness values found in the present study after HF and sandblasting of the materials. Different roughness created by a surface treatment may be attributed to higher ceramic content of Vita Enamic (86 wt% feldspar ceramic) compared with Lava Ultimate (80 wt% nanoceramic). The HF surface treatment modifies the microstructures of CAD/CAM hybrid ceramic surface by partial dissolution of the polymer and glassy phase of the feldspar ceramic, forming microporosity on the ceramic surface.²⁹

The test methods for evaluating the adhesion of resin composites are the shear bond strength test, tensile bond strength test, micro tensile bond strength test, and pull off and push out tests. Each method, in which different calculation is used to determine bond strength value, has inherent advantages and shortcomings.³²⁻³⁴ Higher strength values are generally found with shear bond strength test. However, comparison of bond strength values obtained from different test methods would not be appropriate.³² The shear test is the commonly used test for evaluating the composite repair bonding.^{27,35} Advantages of the shear test are easy specimen preparation and simple test protocol. However, non-uniform stress distribution in the adhesive area should be taken into consideration.³⁶ Another limitation is that the polymerization shrinkage of resin composite cements is not considered in this method.^{32,37} Shear bond strength test is performed by applying the force parallel to the bonding interface³⁸ and the shear bond strength is calculated by dividing the maximum load (in N) to the surface area (in mm^2) of the composite resin. Namely shear bond strength value (in MPa) is the stress on the unit of area. Therefore, the diameter of the composite resin, which is reported in several studies to be ranging from 3 to 6 mm,³⁹⁻⁴¹ does not affect bond strength value.

For clinical applications, 15-25 MPa bond strength for direct composite resin has been reported as an optimal value depending on the composite material and repair method.^{28,42} However, limited data have been published for CAD/CAM resin-ceramic hybrid materials.^{13,27-29} In the present study, Lava Ultimate showed a mean bond strength of 13.42 ± 0.55 MPa (ranging between 2.51-16.63) and Vita Enamic showed a mean bond strength of 17.52 ± 0.44 MPa (ranging between 13.95-21.73), the results which are in agreement with previous studies.^{13,27-29} For GC Cerasmart, however, no data have been published to date. Untreated LU showed the lowest shear bond strength values among the groups. Also, most of the failed specimens were also observed in LU. Lava Ultimate is composed of composite resin material 80% silica and zirconia nanoparticles and nanoclusters by weight.⁹ The zirconia content may have resulted in failure because of the difficulty of polishing. On the other hand, the manufacturer states that using LU for crown restorations is contraindicated. Although it is difficult to define clinically relevant bond strength value after repair for CAD/CAM resin-ceramic hybrid materials, all groups except control groups with no surface treatment

showed approximately 15 MPa bond strength values and consequently could be considered sufficient for clinical application.

Surface treatments are shown to improve the bond strength of resin composite to CAD/CAM resin-ceramic hybrid materials for repair, which is in line with the results of this study.^{13,27,28} In this study, SBS values revealed that untreated control group showed lowest bond strength. Grinding generally showed the highest bond strength values and this finding was in accordance with results of Wiegand *et al.*²⁸ Depending on this finding, grinding the surface with a diamond bur can be recommended as surface pretreatment for bonding composite resin to resin-ceramic hybrid materials; grinding with a diamond bur can be used anywhere quickly and easily. Moreover, in the study, airborne particle abrasion and hydrofluoric acid methods had clinically considerable shortcomings.^{13,29} In contrast to our findings, Stawarczyk *et al.*²⁷ demonstrated that bond strength of resin composite to CAD/CAM resin nanoceramic was significantly lower after grinding compared with air-abrasion with silicized sand. In the present study, although the bond strengths after using airborne particle abrasion were generally lower than grinding and acid-silane application, these values were higher than the groups that had not undergone surface treatment. Surface treatment before repair process should be performed in any case in order to obtain stronger bond between two materials.

Restorations typically fail after being aged in a humid and thermally dynamic oral environment.¹³ Thermal cycling is an artificial aging method of dental materials, and thermal strain on the bonding surface by influence of liquids and thermal change is simulated.⁴³ In the present study, a total of 6 specimens showed failure during thermal cycling, which may indicate the stress and alterations caused by thermal cycling on the repaired surfaces of restorative materials. Analyzing pretest failures revealed that all failures occurred in the control groups with no surface treatment. Under thermal aging, the bond strength is affected by several factors including temperature settings, dwell time, and the number of cycles, in which the latest is the most influential factor.⁴⁴ In this present study, two aging groups were subjected to 10000 thermal cycles, which simulates one year of *in vivo* function.^{45,46} After such a high number of thermal cycling, no significant difference was found between bond strengths of the materials. In previous studies,^{27,28} a CAD/CAM resin nanoceramic (Lava Ultimate) was aged by thermal cycling before adhesive bonding of the resin composite and before bond strength testing to simulate clinical conditions of aging the restoration before failure and aging the restoration after repair. Therefore, the effect of thermal cycling on the bond strength is unclear. In the present study, to assess the effect of aging on bond strength of CAD/CAM resin-ceramic hybrid materials, a control group that was not subjected to thermal cycling was also included in addition to thermal aged specimens before and after resin composite repair. The results showed that thermal cycling did not affect the bond strength between composite resin

and CAD/CAM resin-ceramic hybrid materials except acid and silane applied GC specimens subjected to thermocycling before packing of the composite resin. Previous studies evaluating bond strength of composite resin to CAD/CAM resin-ceramic hybrid materials for intraoral repair were performed only with thermocycled specimens.^{13,27,28} The negative effect of thermocycling on the bond strength of composite resin to CAD/CAM resin-ceramic hybrid restorations were reported previously.^{43,47,48}

One limitation of the present study is the use of a single type of material. It has been reported that the type of composite resin influences its bond strengths to ceramic.⁴⁹ In this study, a microhybrid direct composite resin, which contain barium glass and silica fillers (85.5 wt%), was used as a repair material. This composite resin was selected owing to suitability of the material for anterior and posterior direct restorations as well as intraoral repairs of fractured crowns and bridges. Further studies are needed to evaluate the bond strength of different type of composite resins to CAD/CAM resin-ceramic hybrid materials as several type of composite resins (e.g. nanohybrid, microhybrid, hybrid types with different fillers) are available in the dental market. Another limitation of the present study is the presence of failed specimens before shear bond strength test. In statistical analyses, failed specimens were excluded and shear bond strength data of remaining specimens were analyzed. This should be considered when the results are being evaluated. In addition, long term clinical studies with a large number of patients should be performed on repair durability of CAD/CAM resin-ceramic hybrid materials to provide reliable information for clinicians. On the other hand, the use of artificial saliva as well as thermocycling would ensure closer simulation of clinical conditions.

CONCLUSION

Within the limitations of this study, the following conclusions can be drawn:

Surface treatment methods increased surface roughness of CAD/CAM resin-ceramic hybrid materials. Grinding generated the highest surface roughness among the surface treatment methods. Surface treatment methods improved the bond strength of the composite resin and CAD/CAM resin-ceramic hybrid materials. Thermocycling generally decreased the shear bond strength of the materials. The highest shear bond strength was observed in grinded or acid and silane applied groups while the lowest shear bond strength was observed in untreated groups for all materials in non-thermocycled group.

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