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Effects of surface treatments on repair bond strength of a new CAD/CAM ZLS glass ceramic and two different types of CAD/CAM ceramics

Ayse Seda Ataol¹⁾ and Gulfem Ergun²⁾

¹⁾Department of Prosthodontics, Faculty of Dentistry, Mersin University, Mersin, Turkey

²⁾Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara, Turkey

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Abstract: We evaluated the effects of three different surface treatments and two different bonding procedures on shear bond strength of resin composites to different computer-aided design/manufacturing (CAD/CAM) ceramics with or without thermocycling. The test specimens were prepared from three different CAD/CAM ceramics (Group A, IPS e.max CAD; Group B, IPS e.max ZirCAD; Group C, Vita Suprinity). They were divided into eight subgroups according to the surface treatment and bonding procedures. After resin composite was applied to the surfaces of all test specimens, they were stored in distilled water for 24 h at 37°C. Next, half of the test specimens were subjected to a thermocycling procedure, and the other half were stored in distilled water at 37°C throughout thermocycling. The shear bond strength was performed using a universal testing machine. Statistical analysis revealed that the ceramic types ($P > 0.0031$) and thermocycling ($P > 0.0021$) did not, but the surface treatment technique did significantly affect the shear bond strength values ($P < 0.05$; except group C). Higher bond strength values were observed with HF etching in Groups A and C compared with the other tested surface treatments.

Keywords: CAD/CAM ceramics; repair bond strength; resin composite; bonding agents; surface treatments.

Introduction

Interest has been growing in computer-aided design/manufacturing (CAD/CAM) ceramics due to their simplified manufacturing, superior aesthetic properties, adequate fracture resistance against occlusal forces, and bonding strength between tooth and ceramic surfaces (1,2). Despite the many positive characteristics of ceramic restorations, ceramic fracture is one of the most important causes of restoration loss (3). Therefore, development of mechanical strength and aesthetic properties of ceramics are the primary focuses of dental ceramic developers (4). Generally, mechanical advantages of zirconia ceramics, and aesthetic advantages of glass ceramics, are remarkable. The need to combine the positive properties of these two materials have directed researchers to develop a new generation of glass ceramics (4). Besides the current CAD/CAM blocks (IPS e.max CAD and IPS e.max ZirCAD), a zirconia-reinforced lithium silicate (ZLS) glass ceramic (Vita Suprinity) was presented as a new generation CAD/CAM block in 2013. New generation ZLS glass ceramics differ from the other CAD/CAM glass ceramics due to increased zirconia content (8-12%) and fine-grained homogeneous structure (0.5-0.7 μm) (5), which have been claimed to improve the mechanical properties. Furthermore, the homogeneous dispersion of zirconia in glass phase could provide superior aesthetic properties (6, Vita Zahnfabrik. Vita Suprinity technical and scientific documentation. <http://vitanorthamerica>).

Correspondence to Dr. Ayse Seda Ataol, Department of Prosthodontics, Faculty of Dentistry, Mersin University, Ciftlikkoy Campus, 32133 Street, 33343 Yenisehir, Mersin, Turkey
Fax: +90-32436100-37 E-mail: sedaataol@gmail.com

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Today, glass-ceramic systems that are appropriate for increasing aesthetic expectations of the patient often are preferred for clinical use (7). Therefore, long-term use and maintenance of these systems are important for patients and dentists. Renewal of ceramic restorations, which are fragile and expensive, leads to loss of time and money, and damage to the surrounding tissue. Therefore, repair of a fractured restoration intraorally has clinical importance (8). When the fracture occurs with CAD/CAM blocks, the goal of the methods of fracture repair is to achieve the function and aesthetic of restoration with repair material again (9). Surface treatments are very important to ensure optimal bonding between the resin composite and ceramic surface (3). Micromechanical bonding procedures that could be used include acid etching (etching with hydrofluoric acid [HF], acidulated phosphoric acid, or a phosphate fluoride), and roughening with air abrasion (with aluminum oxide), a diamond bur, or laser (9,10).

Laser irradiation is a relatively safe and easy method for mechanical surface modification on ceramic surfaces (11). Studies have evaluated the effects of laser types, including neodymium:yttrium aluminum garnet (Nd:YAG); erbium (Er):YAG; Er, chromium:yttrium, scandium, gallium, garnet (Er,Cr:YSGG); and carbon dioxide lasers on the bond strength of resin composite to the ceramic surfaces (11,12). Although there are slight differences between the Er:YAG and Er,Cr:YSGG lasers, such as laser wavelength, pulse durations, and energy, they basically are similar in their properties (10). Information about the effects of the Er,Cr:YSGG laser system on the surface treatment of CAD/CAM ceramics is scarce. Because of the rapid increase in use of ZLS glass ceramic CAD/CAM blocks, there is a need to evaluate the effects of Er,Cr:YSGG laser irradiation on this new generation of CAD/CAM blocks and to compare this effect with lithium disilicate glass ceramic CAD/CAM blocks and yttrium-stabilized zirconia ceramic CAD/CAM blocks.

In addition to mechanical surface treatments, another important step in ceramic repair is chemical bonding. Chemical bonding between ceramic surface and hydrophobic resin can be established with phosphate monomers, such as silane, or bifunctional monomers, 3-methacryloxy propyl-trimethoxysilane (MPS), which are found in silane coupling agents (13). They create dual covalent linkages between organic (composite) and inorganic (ceramic) structures. Reducing the application steps is the novel tendency in adhesive dentistry to simplify bonding procedures (14). Some molecules

can create a direct link between metal or oxide ceramic surfaces with resin composite. These structures are available commercially as metal or ceramic primers, which comprise a combination of silane and metal/ceramic primer (15).

Although many studies exist in the literature related to surface treatments that are applicable to feldspathic and leucite ceramics, lithium disilicate glass ceramics, and ceramics that contain high alumina and zirconia (3,8), there is little information related to the new generation ceramic type (6,16). The new generation ZLS glass ceramic has a similar silica content (56-64%) to that of glass ceramics except for increased zirconia content (8-12%) (5). It was thought that increased zirconia content could change the necessary surface treatment for the roughening procedure and bonding agents.

However, information about the repair protocols of the new generation ZLS glass ceramic remains scarce. We evaluated the effects of surface treatments on shear bond strength of resin composites of three different CAD/CAM ceramics (lithium disilicate glass, yttrium-stabilized zirconia, and ZLS glass ceramics) with or without thermocycling (TC+). We hypothesized that the effects of ceramic type on shear bond strength will be significant, the effects of surface treatment on shear bond strength will be positive, and TC+ will decrease the shear bond strength.

Materials and Methods

The codes of the tested ceramic types and detailed compositions of the test materials used in this study are summarized in Table 1. A total of 336 blocks were cut from CAD/CAM blocks ceramics using a water-cooled diamond blade (Diamond Wafering Blade, 15LC, 11-4255, 127 × 0.4 mm; Buehler, Lake Bluff, IL, USA) with a low-speed cutting saw (Isomet 1000; Buehler), cutting liquid, and an anticorrosive (Cool; Buehler). Test specimens were prepared from three different CAD/CAM ceramics (Group A, IPS e.max CAD; Group B, IPS e.max ZirCAD; Group C, Vita Suprinity). Specimens from Groups A and C were prepared with dimensions of 12 × 12 × 3 mm. Next, they were crystallized in a porcelain oven (Programat EP 3000; Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's recommendations. Specimens from Group B were prepared with dimensions of 15 × 15 × 3.75 mm. The shrinkage rate and dimensions of zirconia specimens were determined with a pilot study. According to the manufacturer's recommendations, after the recommended sintering cycle in the sintering furnace (Sirona Dental Systems GmbH, Bensheim, Germany), the Group

Table 1 Materials used in this study

Product	Material	Codes of test groups	Material content	CAD/CAM ceramic block sizes	Lot No.	Manufacturer
IPS e.max CAD, LT A2/C14	Lithium disilicate glass ceramic CAD/CAM blocks	Group A	(SiO ₂ 57-80%, Li ₂ O 11-19%, K ₂ O 0-13%, P ₂ O ₅ 0-11%, ZrO ₂ 0-8%, ZnO 0-8%, others and coloring oxides 0-12%)	12 × 14 × 18 mm	T27280	Ivoclar Vivadent, Schann, Liechtenstein
IPS e.max Zir-CAD MO2 C15	Yttrium-stabilized zirconium oxide ceramic CAD/CAM blocks	Group B	(ZrO ₂ 87-95%, Y ₂ O ₃ 4-6%, HfO ₂ 1-5%, Al ₂ O ₃ 0-1%)	14.5 × 15.5 × 18.5 mm	T29204	Ivoclar Vivadent
Vita Suprinity A2 T-LS-14	Zirconia-reinforced lithium silicate glass ceramic CAD/CAM blocks	Group C	(SiO ₂ 56-64%, Li ₂ O 15-21%, K ₂ O 1-4%, ZrO ₂ 8-12%, P ₂ O ₅ 3-8%, Al ₂ O ₃ 1-4%, CeO ₂ 0-4%, pigments 0-6%)	12 × 14 × 18 mm	51367	Vita Zahnfabrik, Bad Säckingen, Germany
Ultradent Porcelain Etch	9% Hydrofluoric acid				B856T	Ultradent Products Inc, South Jordan, UT, USA
Ultradent Silane	MPS				BBSVP	Ultradent Products Inc
Clearfil Majesty ES-2 (A2)	Light-curing restorative composite resin containing nano-fillers				170032	Kuraray Noritake Dental Inc, Kurashiki, Japan
Clearfil Universal Bond	MDP containing light-curing one-component bonding agent				3D0006	Kuraray Noritake Dental Inc
Clearfil Ceramic Primer	3-MPS, 10-MDP-containing one-component silane coupling agent				930004	Kuraray Noritake Dental Inc

Table 2 Overview of study plan and subgroups

Group A IPS e.max CAD (<i>n</i> = 112)	Group B IPS e.max ZirCAD (<i>n</i> = 112)	Group C Vita Suprinity (<i>n</i> = 112)
1 ST: HF etching + ultradent silane + Clearfil Universal Bond (<i>n</i> = 14)		
2 ST: alumina blasting + ultradent silane + Clearfil Universal Bond (<i>n</i> = 14)		
3 ST: laser + ultradent silane + Clearfil Universal Bond (<i>n</i> = 14)		
4 ST: HF etching + Clearfil Ceramic Primer (<i>n</i> = 14)		
5 ST: alumina blasting + Clearfil Ceramic Primer (<i>n</i> = 14)		
6 ST: laser + Clearfil Ceramic Primer (<i>n</i> = 14)		
7 ST: ultradent silane + Clearfil Universal Bond (control) (<i>n</i> = 14)		
8 ST: clearfil Ceramic Primer (control) (<i>n</i> = 14)		
Thermocycle (TC+) (<i>n</i> /2)		Water storage (TC-) (<i>n</i> /2)

ST: surface treatment.

B specimens reached approximately 12 × 12 × 3 mm as a result of the 20% to 25% sintering shrinkage. All test specimens were measured with a digital caliper (World Precision Instrument, Sarasota, FL, USA). The surface of the specimens was cleaned with a steam cleaner. Specimens were embedded in autopolymerizing acrylic resin using a polyethylene mold (with dimensions of 16 × 16 × 10 mm) so that one surface of the specimens remained uncovered for bonding procedures. The exposed surfaces were flattened under water refrigeration using 600, 800, and 1,200 grit silicon carbide paper (English Abrasives, London, UK) on an automated grinder-polisher device (Gripo 2V Grinder-Polisher; Metkon Instruments, Inc., Bursa, Turkey). Next, the specimens were cleaned

ultrasonically (Bandelin Sonorex; Bandelin Electronic GmbH & Co. KG, Berlin, Germany) in distilled water for 10 min, and dried with oil-free air spray for 15 s. Subsequently, they were divided into eight test groups (*n* = 14 per group; Table 2) according to the three different surface treatment techniques and two different bonding procedures.

The details of surface treatments and application procedures are as follows:

Acid etching: Etched with 9% hydrofluoric acid for 90 s (according to manufacturer's recommendation), rinsed for 30 s, and air dry with oil-free air.

Alumina blasting: Alumina blasting with a device (Rotaks-Dent Dentistry, Inc., Istanbul, Turkey) using 50

$\mu\text{m Al}_2\text{O}_3$ particles (Korox; Bego, Bremen, Germany) for 20 s, applied perpendicularly to the surface at a pressure of 28 pounds per square inch (psi) from a distance of approximately 10 mm.

Laser etching: Output energy of 3 W, air (50%) and water (1%) cooling at 2.94 μm wavelength, pulse repetition of 50 Hz, pulse duration of 140 μs . An Er,Cr:YSGG laser (WaterLase iPlusTM, Biolase Technology, Inc., Irvine, CA, USA) was delivered by a 600 μm diameter and 6 mm length fiberoptic sapphire tip (MGG6-6, Biolase, Inc., Irvine, CA, USA) that was hand-adjusted with a 45° angle to the ceramic surface at a distance of approximately 1 mm.

Silane application: Apply Ultradent silane with a tip and let it evaporate for 60 s. If not completely dry after 60 s, air dry with oil-free air.

Bond application: Apply Clearfil Universal Bond to the ceramic surface with an applicator brush and rub it in for 10 s. Dry the ceramic surface sufficiently by blowing mild air for more than 5 s until the bond does not move. Light-cure the bond with a dental curing unit according to the manufacturer's recommendations.

Primer application: Apply Clearfil Ceramic Primer to the ceramic surface and dry gently with oil-free air.

All specimens were stored in distilled water in an ultrasonic cleaning device for 5 min and dried. The surface treatments and bonding agents were applied. Then, resin composite containing nano-fillers (Clearfil Majesty ES-2) was put onto the treated test specimen surfaces using a thermoplastic night-guard as a mold (4 × 4 × 2 mm). The single-layer resin composite (2 mm) was cured for 6 s from two directions at an angle of approximately 45° and perpendicularly to the surface for a total of 18 s of curing time using a light-emitting diode (LED) light curing unit (Valo, LED; light output, 3,200 mW/cm²; Ultradent Products, Inc., South Jordan, UT, USA). Afterwards, all specimens were stored in distilled water for 24 h at 37°C. The half of the specimens ($n = 7$) were exposed to thermocycling (TC+; SD Mechatronik Thermocycler, Julabo GmbH, FT 200, Seelbach, Germany) between 5°C and 55°C for 5,000 cycles with a 60 s dwell time and 6 s transfer time. Until the thermocycle procedure ended, the other half were stored in distilled water (TC−) at 37°C in an incubator (Kottermann GmbH & Co. KG Labortechnik, Uetze, Germany).

Each specimen was tightened in a metal holder in a universal testing machine (Llyod Universal Testing Machine; AMETEK, Inc., Hampshire, UK). Load was applied parallel to the long axis of the specimen through a wedge at the ceramic-resin composite interface at a crosshead speed of 0.5 mm/min until the resin composite

Table 3 Percent of fracture types occurred with shear bond strength test and determined with optical microscopy

	Group A	Group B	Group C
1 ST			
TC−	Ad (7/7)	Ad (5/7) Mix (2/7)	Ad (6/7) Mix (1/7)
TC+	Ad (4/7) Mix (3/7)	Ad (7/7)	Ad (5/7) Mix (2/7)
2 ST			
TC−	Ad (7/7)	Mix (7/7)	Ad (6/7) Mix (1/7)
TC+	Ad (2/2)	Ad (2/7) Mix (5/7)	Ad (2/2)
3 ST			
TC−	Ad (6/6)	Ad (3/7) Mix (4/7)	Ad (2/2)
TC+	Ad (3/3)	Ad (4/7) Mix (3/7)	*
4 ST			
TC−	Ad (5/7) Mix (2/7)	Ad (5/7) Mix (2/7)	Ad (5/7) Mix (2/7)
TC+	Ad (6/7) Mix (1/7)	Ad (5/5)	Ad (7/7)
5 ST			
TC−	Ad (7/7)	Ad (3/7) Mix (4/7)	Ad (6/7) Mix (1/7)
TC+	Ad (7/7)	Ad (5/7) Mix (2/7)	Ad (6/6)
6 ST			
TC−	Ad (4/4)	Ad (6/7) Mix (1/7)	Ad (4/4)
TC+	Ad (3/3)	Ad (4/5) Mix (1/5)	*
7 ST			
TC−	Ad (2/2)	Ad (1/3) Mix (2/3)	Ad (1/1)
TC+	Ad (1/1)	Ad (2/3) Mix (1/3)	Ad (1/1)
8 ST			
TC−	Ad (4/4)	Ad (4/4)	Ad (4/4)
TC+	Ad (2/2)	Ad (2/2)	Ad (1/1)

*Premature failure was seen during thermocycle in all specimens in Groups C, 3 ST, TC+ and C, 6 ST, TC+. Ad: adhesive failure, Mix: mixed failure, ST: surface treatment.

delaminated. The maximum load at failure of the resin composite was recorded. Shear bond strengths (MPa) were calculated by dividing the failure load (N) by the bonding area (mm²). After the shear bonding test, fractured surfaces were examined using an optical microscope (Leica MZ 12; Leica Microsystems GmbH, Wetzlar, Germany) at ×12.5 magnification to determine failure type. The failure modes were categorized as follows: adhesive failure (Ad; between ceramic and resin composite interface), cohesive failure (Coh; failure inside the resin composite), and mixed failure (Mix; adhesive and cohesive failure). To examine the effects of surface treatments on the surface topography of CAD/CAM ceramics, two more test specimens were prepared from

Table 4 Shear bond strength values according to ceramic type, surface treatment, and thermocycle (independent of the other parameters)

	Shear bond strength (MPa)	<i>P</i> value
Ceramic type		0.090†
A	10.8 (5.5)	
B	9.3 (7.1)	
C	10.5 (4.9)	
Surface treatment		<0.001†
1 ST	13.2 (4.6) ^A	
2 ST	12.2 (3.4) ^A	
3 ST	8.0 (3.6) ^B	
4 ST	12.2 (5.0) ^{AC}	
5 ST	11.2 (3.3) ^C	
6 ST	5.1 (4.0) ^{BD}	
7 ST	2.9 (2.0) ^D	
8 ST	2.5 (1.9) ^D	
Thermocycle		0.253‡
TC–	10.3 (6.3)	
TC+	10.2 (5.9)	

Data are expressed as median (interquartile range). †Kruskal-Wallis test, ‡Mann-Whitney *U* test, *P* value in bold indicates significant difference in Shear bond strength values between surface treatments ($P < 0.05$). No statistically significant difference was determined among the groups shown with the same big letter ($P > 0.05$).

CAD/CAM ceramics. The surfaces of these specimens were sputtered with a gold layer (Sputter Coater SC7620; 10 kV, working distance 12 mm, spotsize 50, signal SEI; Polaron, VG Microtech, UK) and examined with scanning electron microscopy (SEM; JSM-6060LV; Jeol Ltd, Tokyo, Japan) at $\times 200$, $\times 500$, $\times 1,000$, and $\times 2,000$ magnifications.

Statistical analysis

Data were analyzed using statistical software (IBM SPSS v. 17.0; IBM Corporation, Armonk, NY, USA). Whether the distributions of continuous variables were normal or not was determined by the Kolmogorov Smirnov test. The Levene test was used to evaluate homogeneity of variances. Data were shown as median (interquartile range [IQR]). When the number of independent groups was two, the significance of the difference between groups in terms of median bond strength force was investigated by the Mann-Whitney *U* test. Additionally, the Kruskal-Wallis test was applied for comparisons among more than two independent groups. When the *P* value from the Kruskal-Wallis test statistics was statistically significant, Conover's multiple comparison test was used. Unless otherwise indicated, $P < 0.05$ was considered statistically significant. In our study, for all possible multiple comparisons, the Bonferroni correction was applied for controlling Type I error.

Table 5 Shear bond strength values according to surface treatment (independent of the effects of ceramic type and thermocycle)

	Group A	Group B	Group C
TC–			
1 ST	14.4 (6.4) ^{a,b,c,d}	10.9 (5.5) ^{a,b,c,d}	15.8 (2.9)
2 ST	12.8 (1.9) ^{e,f,g}	14.7 (4.2) ^{e,f,g,l}	10.5 (6.3)
3 ST	9.7 (2.8) ^a	6.3 (3.0) ^{a,l,m,n}	9.6 (0.5)
4 ST	13.2 (8.6) ^{b,i,j}	11.0 (6.6) ^{b,i,j,m}	12.7 (11.6)
5 ST	11.6 (4.2) ^k	12.0 (1.8) ^{k,n,o,p}	10.6 (3.9)
6 ST	5.2 (2.6) ^{b,e,h}	5.5 (4.2) ^{b,e,h,o}	9.5 (1.5)
7 ST	2.5 (1.7) ^{c,f,i}	3.2 (4.0) ^{c,f,i,p}	2.8 (0.0)
8 ST	2.3 (2.0) ^{d,g,j,k}	3.2 (4.4) ^{d,g,j,k}	2.7 (2.0)
<i>P</i> value†	<0.001	<0.001	0.011
TC+			
1 ST	12.0 (6.2) ^{b,c,d,r}	10.2 (4.2) ^{b,c,d}	13.6 (3.6)
2 ST	11.6 (1.1) ^{e,g}	11.4 (3.3) ^{e,f,g,l}	10.1 (0.5)
3 ST	8.1 (1.2)	6.2 (4.4) ^{l,m}	–
4 ST	12.9 (4.9) ^{b,i,j,s}	11.4 (3.5) ^{b,i,j,m}	12.0 (5.1)
5 ST	8.7 (1.3) ^{t,s}	11.2 (8.6) ^{k,o,p}	10.6 (5.4)
6 ST	3.5 (1.7) ^{b,e,h}	5.0 (1.9) ^{b,e,h,o}	–
7 ST	1.5 (0.0) ^{e,i}	3.6 (1.2) ^{c,f,i,p}	1.2 (0.0)
8 ST	1.6 (0.5) ^{d,g,j}	3.8 (0.3) ^{d,g,j,k}	1.6 (0.0)
<i>P</i> value†	<0.001	<0.001	0.040

†Kruskal-Wallis test, according to the Bonferroni Correction, the results were considered statistically significant for $P < 0.0083$. The same superscript letters indicate statistically significant differences between surface treatments. a: 1 ST and 3 ST ($P < 0.0083$), b: 1 ST and 6 ST ($P < 0.0083$), c: 1 ST and 7 ST ($P < 0.0083$), d: 1 ST and 8 ST ($P < 0.0083$), e: 2 ST and 6 ST ($P < 0.0083$), f: 2 ST and 7 ST ($P < 0.001$), g: 2 ST and 8 ST ($P < 0.0083$), h: 4 ST and 6 ST ($P < 0.0083$), i: 4 ST and 7 ST ($P < 0.0083$), j: 4 ST and 8 ST ($P < 0.001$), k: 5 ST and 8 ST ($P < 0.0083$), l: 2 ST and 3 ST ($P < 0.001$), m: 3 ST and 4 ST ($P < 0.0083$), n: 3 ST and 5 ST ($P = 0.002$), o: 5 ST and 6 ST ($P < 0.0083$), p: 5 ST and 7 ST ($P < 0.0083$), r: 1 ST and 5 ST ($P = 0.002$), s: 4 ST and 5 ST ($P < 0.001$).

Results

Because of the comprehensive study design, in the Results and Discussion sections, the names of the test groups were abbreviated as noted in Table 2.

During aging with thermocycling, premature failures (debonding) were observed in the laser groups in Group C, which were tested with two different bonding procedures (3 and 6 ST, TC+). The premature failures occurred on the second and third days of the thermocycle process (Table 3). These groups were excluded from the statistical evaluation because the premature failures occurred before the shear test was applied.

In our study, the shear bond strength was evaluated statistically according to ceramic type, surface treatment, and thermocycling (Table 4). In these analyses, generally, there were statistically significant differences among the test groups in terms of surface treatments ($P < 0.05$). However, ceramic types and thermocycling had no statistically significant effect on shear bond strength

Fig. 1 Representative optical micrographic images of the typical failure types for each tested ceramic. (a) Representative adhesive failure image of Group A (black arrow indicates residual bonding agent around the bonding area, white arrow indicates clear lithium disilicate glass ceramic surface exposed after adhesive failure). (b) Representative mixed failure image of Group B (black arrow indicates residual resin composite on the bonding area, white arrow indicates clear yttrium-stabilized zirconia ceramic surface exposed after mixed failure). (c) Representative adhesive failure image of Group C (black arrow indicates residual bonding agent around the bonding area, white arrow indicates clear ZLS glass ceramic surface exposed after adhesive failure).

Fig. 2 SEM images of CAD/CAM ceramics in (a) Group A-(polished), (b) Group A-(HF etching), (c) Group A-(50- μ m alumina blasting), (d) Group A-(Er,Cr:YSGG laser), (e) Group B-(polished), (f) Group B-(HF etching), (g) Group B-(50- μ m alumina blasting), (h) Group B-(Er,Cr:YSGG laser etching), (i) Group C-(polished), (j) Group C-(HF etching), (k) Group C-(50- μ m alumina blasting), and (l) Group C-(Er,Cr:YSGG laser etching).

values ($P > 0.05$). This result could be related to the large number of variables and subgroups. Therefore, multiple comparison tests with Bonferroni correction were applied (Table 5).

The percent of fracture types that occurred with the shear bond strength test and fractures as determined by optical microscopy (Fig. 1) are shown in Table 3. No statistically significant difference was observed among the ceramic types in terms of the shear bond strength values (independent of the effect of surface treatment and thermocycling; $P > 0.0031$).

In addition, thermocycling had no statistically significant effect on shear bond strength in any test groups compared with water storage groups (TC-; $P > 0.0021$).

In Groups A and B, statistically a significant difference was seen among different surface treatments in terms of shear bond strength (independent of the effect of ceramic type and thermocycling; $P < 0.0083$; Table 5). However, in Group C, no statistically significant difference was observed among different surface treatments ("Group C, TC-," $P = 0.011$ and "Group C, TC+," $P = 0.040$; Table 5).

In Group A, HF etching showed higher shear bond strength values than the other test groups. However, generally, in Group A (except 4 ST and 5 ST of Group A, TC+), there were no statistically significant differences between HF etching and alumina blasting, independent of the bonding procedures applied ($P > 0.0083$). Addi-

tionally, shear bond strength values of laser etching were statistically lower than those of HF etching ($P < 0.0083$). Although laser etching displayed lower shear bond strength values than alumina blasting, no statistically significant differences were seen between them ($P > 0.0083$). In Group B, alumina blasting represented higher shear bond strength values than the other test groups. However, in Group B, there was no statistically significant difference between HF etching and alumina blasting, independent of the bonding procedures performed ($P > 0.0083$). Moreover, shear bond strength values of laser etching were statistically lower than the values of HF etching and alumina blasting in Group B, TC- ($P < 0.0083$). Additionally, in Group B, there was no statistically significant difference between laser etching and control groups ($P > 0.001$). In Group C, although the highest shear bond strength values were observed with HF etching, there were no statistically significant differences among the tested surface treatments and bonding agents ($P = 0.04$).

All of our results revealed that shear bond strength values were not related to the bonding procedures. Thus, the effects of surface treatments on surface topography of the tested CAD/CAM ceramics were examined with SEM (Fig. 2). In Groups A and C, the SEM analyses demonstrated that HF etching created micro porosity but alumina blasting demonstrated an irregular surface (Fig. 2b, c, j, k). In Group B, the effects of alumina blasting were more effective than those of HF etching (Fig. 2g, f). In all three tested ceramic types, the effectiveness of laser etching was much lower than that of alumina blasting and HF etching (Fig. 2d, h, l).

Discussion

Due to the lack of adhesive material defined as a standard protocol for the repair of ceramic restorations, it has become a routine procedure to use the present bonding systems with different combinations. Chemical steps of bonding comprise a silane coupling agent and bond application (15). In recent years, 10-methacryloxy decyl dihydrogen phosphate (MDP) monomer formulation, which is increasingly preferred, has shown an outstanding bonding success (17). 10-MDP is found in some bonding agents and some primers (18). The primers containing functional monomers, such as 10-MDP are recommended to improve the resin and ceramic bonding (19,20).

In our study, to roughen the surface of the CAD/CAM ceramics, acid etching, alumina blasting, and laser etching have been applied. In addition, silane (Ultradent)/bond (Clearfil universal bond) and an alternatively produced primer (Clearfil Ceramic Primer) were compared. The

bond and primer containing MDP are advised for use in the ceramic repair process. The manufacturer states that Clearfil Ceramic Primer provides a successful bonding between resin composite and ceramic without using a bonding agent. Clearfil Ceramic Primer can reduce clinical practice steps more than routine silane/bond application. In a previous study (21), researchers also reported that no statistically significant differences were observed between the application of silane only and the use of silane followed by bond application. Similarly, in our study, using only a primer or silane/bond application caused no statistically significant difference in the bond strength ($P > 0.0083$). The single-step bonding procedure with primer application presented similar repair bond strength with silane/bond application for the repair of CAD/CAM ceramics. This result could be related to the effectiveness of the enhanced bonding systems and structure of silane (Clearfil Ceramic Primer) containing MDP monomer. Considering these results, a single-stage bonding agent (only Clearfil Ceramic Primer application) can be preferred to silane/bond application, since it not only saves time but it also is easy to use and apply for repairing the fractured CAD/CAM ceramics.

Generally, roughening with a diamond bur, acid etching, alumina blasting, and laser etching are the surface-roughening processes used commonly in studies that test the intraoral repair process *in vitro* (11,15). Studies related to intraoral repair of ceramics by acid etching and alumina-blasting processes are available in the dental literature (8,15). However, the data related to the efficiency of roughening by laser etching are limited (10,11).

In Group A, HF etching followed by silane/bond or primer application demonstrated a statistically higher bond strength than laser etching followed by primer application and in control groups ($P < 0.0083$). HF acid etching creates dissolution acting on the glass phase and provides microporous retention via ensuring the release of crystal field. This micro porosity increases the surface area and provides micromechanical locking with resin structure (22). Previous studies (12,23) reported that the highest bond strength value between glass ceramic and resin cements was obtained with HF etching. In addition, although Kursoglu et al. (12) reported that irradiation at 6 W Er,Cr:YSGG laser application may not be an effective surface treatment type, 1.5 and 2.5 W Er,Cr:YSGG laser application increased the bonding strength more than an untreated ceramic surface. Additionally, a previous study reported the efficiency of HF etching and Er:YAG laser with different energy parameters on the Empress 2. This study indicated that low-energy level laser applica-

tion, although not statistically significant, demonstrated higher bond strength than HF etching (11). Conversely, in our study, the efficiency of 3 W laser application revealed a statistically significant lower bond strength than HF etching in group A ($P < 0.001$). SEM analysis exhibited significant differences in terms of the effects of HF and laser etching in Group A (Fig. 2b, d). Our results may illustrate that the increase in surface roughness formed with HF etching provided an increase in the bond strength.

Borges et al. (24) assessed the effects of HF etching and 50- μm alumina blasting on the IPS Empress and IPS Empress 2 ceramic surfaces. They concluded that the increases in surface roughness with HF etching and alumina blasting were similar. However, in our study, as seen in SEM analysis, HF etching created micro porosity while alumina blasting created an irregular surface topography on the surface in Group A (Fig. 2b, c).

In subgroups of Group A in which Er,Cr:YSGG laser etching was applied, all fracture types were adhesive fractures and also the number of premature failures was higher than in the other groups. However, HF etching subgroups of Group A demonstrated mixed fractures in addition to adhesive fractures. These results indicated that the laser etching created weaker shear bond strength than the other tested surface treatments.

Glass ceramics are more susceptible to acid etching than zirconia ceramics (14). The silica content of ZLS glass ceramic (56-64%) is similar to that of the other glass ceramics. Therefore, this material is considered susceptible to HF etching, unlike zirconia ceramics (6). On the other hand, the increased rate of zirconia (8-12%) in ZLS glass ceramics for strengthening glass ceramics awakens the idea that sensitivity of ZLS ceramics to surface treatments may vary in line with glass ceramics. In all subgroups of Group C (TC- and TC+), there was no statistically significant difference among the different surface treatments ($P > 0.0083$). In our study, although no statistically significant differences were found among the tested subgroups of Group C, the highest bond strength values (Group C, 1 ST, TC-: 15.8 MPa; Group C, 4 ST, TC-: 12.7 MPa) were observed with HF etching. The repair bonding strength of these ceramics was improved by HF etching regardless of the adhesive procedures. However, the bond strength values were lower than in previous studies (6,12,24). Sato et al. (6) reported that, as the HF etching time increased from 20 to 40 s, the bond strength values of ZLS glass ceramics to resin cement decreased due to the exposure of lithium silicate crystals and zirconia particles. Similarly, in our study, the decrease in bond strength values may be related to the

HF etching time (90 s) that creates a surface with lower wettability.

All specimens in the laser etching groups (Group C: 3 ST, TC+ and Group C: 6 ST, TC+) were debonded during thermocycling (Table 3). Thus, despite the decline in bond strength of the alumina-blasted and HF-etched groups, the laser-etched test specimens were most vulnerable to temperature changes in the water, with great loss of bonding during thermocycling. The possible explanation for this finding is ineffectiveness of laser etching on the surface of ZLS glass ceramics, which can be attributed to the increased zirconia content, (8-12%) compared to lithium disilicate glass ceramics (Fig. 2l). Owing to the fact that premature fractures that occurred during thermocycling were not included in the statistical analysis (Table 3), statistical analysis of these groups was performed with a small number of data. This condition could be thought to be a reason why no statistically significant differences were found among the examined groups.

In our study, for the water storage subgroups of Group C (TC-), there were no statistically significant differences among the effects of laser etching followed by silane/bond application, HF acid etching, and alumina blasting groups on the shear bond strength values ($P > 0.0083$), similar to Group A. These results supported that using silane/bond application would be more appropriate when laser etching will be preferred for ZLS glass ceramics. However, the effects of different surface treatments on ZLS glass CAD/CAM ceramics are not the current research topics, particularly for laser etching. Furthermore, the effects of silanization of ZLS glass CAD/CAM ceramics on bonding performance should be investigated further.

The effectiveness of the surface treatments was similar between Groups A and C as shown in SEM analysis. When the fracture types were examined, the ratio of adhesive/mixed failure in Group C (8/1) was similar to that in Group A (11.6/1). However, this ratio was different from the ratio in Group B (1.7/1; Table 3). These results showed that the bonding type of ZLS ceramics was similar to that of lithium silicate glass ceramics (Fig. 1a-c).

In Group B, the highest shear bond strength values were obtained with alumina blasting (Group B, 2 ST, TC-: 14.7 MPa; Group B, 5 ST, TC-: 12.0 MPa). Alumina blasting followed by silane/bond application and HF etching followed by primer application revealed only a significantly higher bond strength than laser followed by primer application and in the control groups ($P < 0.001$). However, HF etching groups showed a

higher bond strength than laser and control groups. Many studies revealed that bonding agents containing 10-MDP functional monomer provide better bonding to oxide ceramics, such as zirconia (25,26). These results may be associated with increased wettability of the surface or the use of primer and bonding agents containing MDP. An *in vitro* study conducted by Borges et al. (24), in which the effects of HF and 50- μ m aluminum oxide on the InCeram zirconia surface were evaluated, revealed that HF did not create any changes in the microstructure of ceramic. The fact that the only phase that could be etchable is the silica phase and that there is no glass phase in the zirconia oxide ceramics can explain this situation (27). However, Derand and Derand (27) reported that HF etching could change the bonding capacity of ceramics or the free energy potential. In this study, the SEM analysis of the yttrium-stabilized zirconium oxide CAD/CAM ceramics that were treated with HF etching revealed that the efficiency of HF etching was low. In addition, alumina blasting (Fig. 2g) provided a significantly higher surface roughness than HF (Fig. 2f) and laser etching (Fig. 2h). Although there was no increase in surface roughness, alumina blasting followed by silane/bond application and HF etching followed by two different adhesive procedures revealed higher shear bond strength values than laser followed by primer application and the control groups. It is claimed that this monomer increases the bonding efficiency of resin composites to the zirconium oxide surface by the manufacturer. Attia et al. (28) reported on the efficiency of a new generation primer that is marketed with the claim that it increases the bond strength of resin composite to the zirconia ceramic surface more than conventional silanes. Another *in vitro* study conducted by Akhavan Zanjani et al. (10), in which alumina blasting and different type of lasers were performed for roughening the surface of zirconium oxide ceramics, revealed that alumina blasting provided the highest bond strength. This result suggested that laser etching creates less surface roughness than alumina blasting, similar to our study.

In Groups A and C, SEM analyses revealed an irregular surface for the alumina-blasted subgroups. This could be related to the alumina particles used for alumina blasting and low surface hardness of these test specimens compared with zirconia ceramics. Although alumina blasting affected different degrees of the surfaces of CAD/CAM ceramics tested in this study, the test surface in Group B was more uniform than that in the other groups. Therefore, alumina blasting obviously is more beneficial for repair of zirconia ceramics than the other groups.

When the fracture types were examined, the ratio of adhesive/mixed failure of Groups B, 1 and 4 ST (5.5/1) and Groups B, 3 and 6 ST (1.8/1) showed a higher mixed failure ratio than Groups B, 2 and 5 ST with the adhesive/mixed ratio of 0.7/1 (Table 3). These results demonstrated the efficiency of alumina blasting on the test specimens of Group B compared with the other tested surface treatments. The 3 W Er,Cr:YSGG laser etching demonstrated quite low shear bond strength values than the other tested surface treatments in Group B of our study. This result was consistent with a previous study, where the investigators reported that alumina-blasted surfaces had higher bond strength than 3 W Er,Cr:YSGG laser application (10).

Overall results of our study indicated that the surface treatments affected the shear bond strength values positively. However, ceramic types and thermocycling did not affect the shear bond strength significantly. Thus, the null hypothesis of this study could be rejected in terms of the effects of ceramic type and thermocycling.

Previous studies (29,30) suggested that aging by thermal cycling decreases the bond strength between ceramics and resins. However, there is no consensus related to the idea that water storage and thermocycling reduce bonding strength between resin and ceramics (8).

Wegner et al. (31) reported that the chemical bonding procedure containing MDP could eliminate the negative effects of thermocycling. Similarly, in our study, decreased bond strength was seen in all thermocycled (artificially aged) groups, but there was no significant difference ($P > 0.0021$). In our study, we examined thermal cycling for 5,000 cycles with two different adhesive procedures that contain MDP. These results are in accordance with the fact that MDP displays stability against hydrolysis during aging as it contains a long carbonyl chain (29). In addition, the mechanism of aging, sensitivity of resin-based material against water absorption depends on the monomer conversion, filler ratio, volume ratio of nano-sized gaps, and amount of polymer crosslinking (32). The nanohybrid composites preferred in our study have less interparticle gaps between the particles than the micro hybrid composite resins (33). This may reduce water absorption of composite resins during thermocycling. Additionally, although thermocycling did not affect the shear bond strength significantly, the premature debondings during thermocycling could be related to the ineffectiveness of the tested surface treatments (especially in Groups C, 3 ST, TC+ and C, 6 ST, TC+).

Failure modes on the bonding surface between tested ceramics and the resin composites reflected the results of

the shear bond strength (Table 3). In most tested groups, adhesive failures were observed at the ceramic-resin composite interface before and after thermocycling. The results of failure mode support the low shear bond strength between the tested ceramics and resin composite was low.

The limitation of this study is that only the repair capability of the fracture type, in which one type of ceramic was exposed on the fracture area, was tested. In our point of view, further research should evaluate the repair bond strength of resin composites to the fracture area in which two types of ceramics were exposed to the fracture area on bilayered all ceramic restorations.

Within the content of our study, the following points can be concluded: 1. Alumina blasting and HF etching provided satisfactory repair bond strengths for each CAD/CAM ceramic tested in this study; 2. The surface treatment of CAD/CAM yttrium-stabilized zirconia ceramics by alumina blasting was more effective than HF etching and Er,Cr:YSGG laser irradiation; 3. The surface treatment of CAD/CAM ZLS and lithium disilicate glass ceramics by HF etching was more effective than alumina blasting and Er,Cr:YSGG laser irradiation; 4. Er,Cr:YSGG laser irradiation could not be regarded as an effective surface treatment for roughening each CAD/CAM ceramic surface to establish better bond strength with a resin composite; 5. A single-stage bonding agent (only Clearfil Ceramic Primer application) may be preferred to silane/bond application for repairing the fractured CAD/CAM, since it not only saves time but it also is easy to use and apply.

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Conflict of interest

None declared.

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