



# Effect of surface treatments on the bond strength of indirect resin composite to resin matrix ceramics

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**PURPOSE.** The purpose of this study was to evaluate the shear bond strength (SBS) of an indirect resin composite (IRC) to the various resin matrix ceramic (RMC) blocks using different surface treatments. **MATERIALS AND METHODS.** Ninety-nine cubic RMC specimens consisting of a resin nanoceramic (RNC), a polymer-infiltrated hybrid ceramic (PIHC), and a flexible hybrid ceramic (FHC) were divided randomly into three surface treatment subgroups (n = 11). In the experimental groups, untreated (Cnt), tribochemical silica coating (Tbc), and Neodymium-Doped Yttrium Aluminum Garnet (Nd:YAG) laser irradiation (Lsr) with 3 W (150 mJ/pulse, 20 Hz for 20 sec.) were used as surface treatments. An indirect composite resin (IRC) was layered with a disc-shape mold (2 × 3 mm) onto the treated-ceramic surfaces and the specimens submitted to thermal cycling (6000 cycles, 5 - 55°C). The SBS test of specimens was performed using a universal testing machine and the specimens were examined with a scanning electron microscope to determine the failure mode. Data were statistically analyzed with two-way analysis of variance (ANOVA) and Tukey HSD test ( $\alpha=.05$ ). **RESULTS.** According to the two-way ANOVA, only the surface treatment parameter was statistically significant ( $P<.05$ ) on the SBS of IRC to RMC. The SBS values of Lsr-applied RMC groups were significantly higher than Cnt groups for each RMC material, ( $P<.05$ ). Significant differences were also determined between Tbc surface treatment applied and untreated (Cnt) PIHC materials ( $P=.039$ ). **CONCLUSION.** For promoting a reliable bond strength during characterization of RMC with IRC, Nd:YAG laser or Tbc surface treatment technique should be used, putting in consideration the microstructure and composition of RMC materials and appropriate parameters for each material. [J Adv Prosthodont 2019;11: 223-31]

**KEYWORDS:** Ceramics; Composite resins; Laser; Shear strength

## INTRODUCTION

In today's dentistry, with the developing technology and progressive material science, Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) technique has become more popular for restorative dentistry because of many superiorities like improved conformity, homogeneity, integrity, less time- and cost- consuming production process compared to conventional techniques.<sup>1-6</sup>

In CAD/CAM processing technologies, ceramic restorations are fabricated by milling of industrial blocks. Based on the standardized industrial fabrication of these blocks, advanced physical and mechanical properties,<sup>6-9</sup> fewer discolorations,<sup>10-12</sup> and higher abrasion resistance<sup>13,14</sup> can be achieved. Besides the various type of ceramic materials (feldspathic, reinforced glass ceramics, zirconia), novel ceramic blocks namely Resin Matrix Ceramics (RMC) have been also improved for CAD/CAM technique.<sup>15,16</sup> Especially, RMC materials have been developed to combine the physical and mechanical advantages of ceramics and improved flexural properties and low abrasiveness of composite resins.<sup>4,17-19</sup> The resin nanoceramic (RNC, Lava Ultimate, 3M ESPE, St. Paul, MN, USA), polymer-infiltrated hybrid ceramic (PIHC, Enamic, Vita Zahnfabrik, Bad Säckingen, Germany), and flexible hybrid ceramic (FHC, Cerasmart, GC Corporation, Tokyo, Japan) materials are popular resin matrix ceramic CAD/CAM blocks in the markets.<sup>17,19-23</sup> Despite their higher physical, mechanical properties and wear resistance, the RMC restorations may be weak on color reproduction and

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aesthetic characterization when fabricated by CAD/CAM milling process on the anterior area.<sup>23,24</sup> Nonfunctional facial and/or incisal areas of CAD/CAM made RMC restorations will be veneered with a resin-based restorative or coating material to gain better aesthetic results.<sup>23-26</sup> Therefore, a combination technique for CAD/CAM made RMC restorations with an indirect resin composite (IRC) material will be suitable for this fabrication process, especially in the anterior region. This technique offers not only better mechanical properties with RMC base, but also improved, more attractive optical properties with IRC veneer supplement.<sup>24,27</sup> However, little information is available on the bonding performance between the RMC and IRC in the case of such combination techniques.<sup>23,28</sup> To increase the bond strength of IRC material to RMC, different surface treatments are used, such as chemical etching with hydrofluoric (HF) acid, acidulated phosphate fluoride, or phosphoric acid, airborne particle abrasion with aluminum oxide particles, tribochemical silica coating, and, in recent years, laser treatment.<sup>2,29-33</sup> Nevertheless, there is still no definitive information about which commercially available method is suitable and effective for the bonding process of indirect resin composite to RMC.<sup>29,34</sup>

Based on these considerations, the purpose of this study was to evaluate the shear bond strength (SBS) of an IRC to the different RMC blocks using different surface treatments. The first null hypothesis was that the different surface treatment applications would not affect the SBS of an IRC to RMC blocks. The second null hypothesis was that the SBS values would not vary depending on the type of RMC materials.

## MATERIALS AND METHODS

This study evaluated the SBS of three different CAD/CAM RMC blocks (PIHC, RNC, FHC), with different surface treatment methods of conditioning for characterization with IRC. Manufacturers and material compositions are presented in Table 1.

Ninety-nine cubic specimens in dimensions of 6 × 6 × 2 mm were prepared from RMC CAD/CAM blocks under water cooling with a cutting machine (Mecatome T180, Presi Metallography, Eybens, France). The dimensions of specimens were measured by a digital micrometer (Digimatic Caliper, Mitutoyo MC, Aurora, IL, USA). The specimens that illustrate differences in size were adjusted at required dimensions. Then all specimens were embedded in autopolymerizing acrylic resin blocks (Meliodent, Heraeus Kulzer, Armonk, NY, USA). Bonding surfaces were polished under water cooling by 200, 400, and 600-grit silicon carbide papers (3M ESPE, St. Paul, MN, USA), respectively. Thereafter, all polished specimens cleaned with an ultrasonic cleaner (Erosonic Energy, Euronda, Vincenza, Italy) for 5 min in distilled water, and air-dried before surface treatments.

The RMC specimens were randomly divided into three subgroups (n = 11) according to different surface treatments. Details of the surface treatments are described in Table 2. After the application of surface treatments, the specimens were cleaned with an ultrasonic cleaner (Erosonic Energy, Euronda, Vincenza, Italy) in distilled water for 10 minutes, and then one sample for each of the nine subgroups was examined with SEM × 2000 magnification (Nova Nano-SEM 450, FEI Comp., Hillsboro, OR, USA).

**Table 1.** Materials used in this study

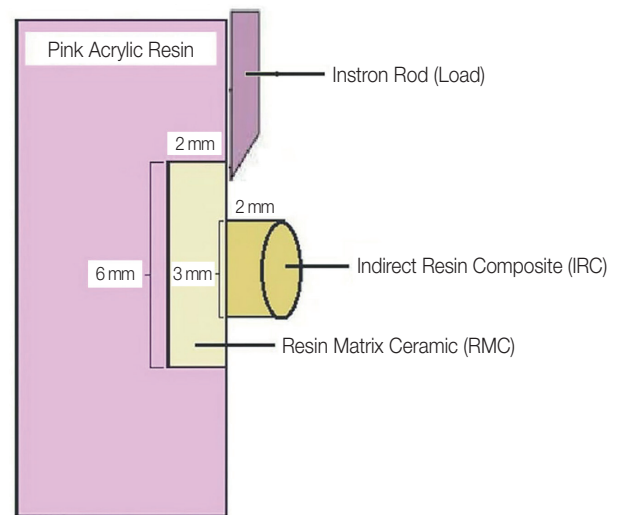
Material	Type of RMC	Composition	Manufacturer
Vita Enamic	Polymer-infiltrated hybrid ceramic (PIHC) (Glass-ceramic in a resin interpenetrating matrix)	86 wt% feldspathic ceramic enriched with Al <sub>2</sub> O <sub>3</sub> , 14 wt% polymer (UDMA, TEGDMA)	Vita Zahnfabrik, Bad Säckingen, Germany
GC CeraSmart	Flexible hybrid ceramic (FHC)	Nanoparticle-filled resin (UDMA, DMA) containing 71 wt% silica and barium glass filler	GC Corp., Tokyo, Japan
Lava Ultimate	Resin nano-ceramic (RNC)	80 wt% nanoceramic, 20 wt% resin (Bis-GMA, UDMA, Bis-EMA, TEGDMA)	
Rely X Ceramic Primer	Ceramic primer	Stabilized ethyl alcohol, MPS	3M ESPE, St. Paul, MN, USA
Single Bond Universal	Adhesive resin	MDP Phosphate Monomer, DMA, HEMA, Vitrebond copolymer, filler, ethanol, water, silane	
CoJet System	Silica-coated agent	50- $\mu$ m silica-coated Al <sub>2</sub> O <sub>3</sub> airborne particles	
Solidex	Indirect composite resin	Micro-hybrid composite containing of over 53 % ceramic filler	SHOFU Inc., Kyoto, Japan

Al<sub>2</sub>O<sub>3</sub> = Aluminium trioxide; UDMA = Urethane dimethacrylate; TEGDMA = Triethylene glycol dimethacrylate; DMA = Dimethacrylate; Bis-EMA = Bisphenol-A-ethoxylate glycidyl methacrylate; Bis-GMA = Bisphenol-A-glycidylmethacrylate; MPS = Methacryloxypropyltrimethoxy silane 2; HEMA = Hydroxyethylmethacrylate; MDP = methacryloyloxy-decyl-dihydrogen-phosphate; FHC, flexible hybrid ceramic; PIHC, polymer-infiltrated hybrid ceramic; RMC, resin matrix ceramics; RNC, resin nanoceramic

**Table 2.** Surface treatment groups

Group	Surface treatment method
Cnt	No surface treatment
Tbc	Tribochemical silica coating with 50- $\mu$ m silica-coated $Al_2O_3$ airborne particles (CoJet System, 3M ESPE, St. Paul, MN, USA) for 10 sec at 2 bar pressure from a distance of 10 mm using an intraoral sandblaster.
Lsr	1064 wavelength Nd:YAG laser (Smartfile, Deka, Firenze, Italy) irradiation at energy settings of 3 W (150 mJ/pulse and 20 Hz) for 20 sec with a pulse duration of 50 $\mu$ s by a non-cooled handpiece with 300 $\mu$ m optical fiber.

Nd: YAG, Neodymium-Doped Yttrium Aluminum Garnet

**Fig. 1.** Schematic view of SBS testing.

Before condensing the IRC, a methacryloxypropyltrimethoxysilane 2 (MPS) containing agent (Rely XTM Ceramic Primer, 3M ESPE, St. Paul, MN, USA) was applied onto the RMC specimens for 60 sec and lightly air-dried. Then, an adhesive material (Scotchbond Universal, 3M ESPE, St. Paul, MN, USA) was applied and light polymerization was performed using an LED light-curing unit (Blue-phase; Ivoclar Vivadent, Liechtenstein) for 20 sec. Finally, an IRC (Solidex, SHOFU Inc., Kyoto, Japan) was incrementally layered onto the treated RMC surfaces using a silicone mold with a disc-shape cavity (2  $\times$  3 mm) to standardize the dimensions of the composite and final polymerization was performed with a light-curing unit (Solidilite V, SHOFU Inc., Kyoto, Japan), which had 4 halogen lamps to uniform polymerization in a curing time of 5 min at the wavelength spectrum of 400 - 550 nm. The application and polymerization of adhesives and composite materials were performed according to the manufacturer's instructions. All specimens were stored in distilled water at 37°C for 24 h to allow post-polymerization. Before the SBS test, the specimens were aged with 6000 cycles of thermocycling between 5°C to 55°C with 30 sec dwelling time and 10 sec transfer time in distilled water.

A universal test machine (Autograph AGS X, Shimadzu Co., Kyoto, Japan) was used for the SBS test. According to the working principle of the universal test machine, specimens were placed in a special device and a moment-free axial force application at 1 mm/min crosshead speed was applied by a knife-edge shaped apparatus between the specimens and resin interface until failure occurred (Fig. 1). The maximum load data were recorded at the time of the failure of the RMC and composite materials. The SBS was specified by dividing maximum failure load by the composite resin surface area ( $a = P/A = N/mm^2$ ) and it was calculated in megapascals (MPa). After the SBS test was completed, the failure modes of all specimens were viewed by an optical microscope (MP 320; Carl Zeiss, Oberkochen, Germany) at

50  $\times$  magnification. The failure types were divided into three different categories as adhesive (between the RMC and IRC interface), cohesive (within the RMC or IRC), and mixed.

In the statistical analyses, the distribution of the data was evaluated using the Shapiro Wilk test and the data showed normal distribution ( $P = .066$ ). Then, Levene's test of homogeneity was utilized. The SBS results were then analyzed by two-way ANOVA to assess the effects of surface treatment, RMC type, and interactions. Also, mean SBS values were compared using Tukey's multiple comparison test ( $\alpha = .05$ ). The failure modes were analyzed with Pearson Chi-Square test and the correlation between SBS and Fracture modes also compared with Kendall's tau\_b correlation analyses. All calculations were performed using SPSS 20.0 V statistical software (SPSS 20.0 V, SPSS Inc., Chicago, IL, USA) and significance was evaluated at  $P < .05$  for all tests.

## RESULTS

The SBS test results of Cnt groups were compared with the Tbc and Lsr groups. According to the two-way ANOVA, only surface treatment parameter was found statistically significant ( $P < .05$ ) on the SBS of IRC to RMC (Table 3). The mean SBS values, standard deviations (SD) and the statistical differences between RMC and surface treatment groups are listed in Table 4. These statistical differences are shown in the table in different letters in terms of surface treatment applications for each RMC group.

The SBS values of Lsr surface treatment applied RMC groups (19.09 - 19.69) were significantly higher than Cnt groups (14.15 - 15.35) for each RMC material ( $P < .05$ ). Additionally, a significant difference was determined between Tbc surface treatment applied and untreated (Cnt) PIHC groups ( $P = .039$ ). There was no significant differ-

**Table 3.** Results of two-way ANOVA test

Variable (source)	Sum of squares	df	Mean squares	F	P
Resin matrix ceramic	13,095	2	6,548	1,900	.156
Surface treatment	335,843	2	167,921	48,725	.000
Interaction	29,716	4	7,429	2,156	.081
Error	279,152	81	3,446		
Total	26562,512	90			

\*Significantly different at  $P < .05$ .

**Table 4.** Mean and SD of SBS values and differences between RMC/surface treatment groups

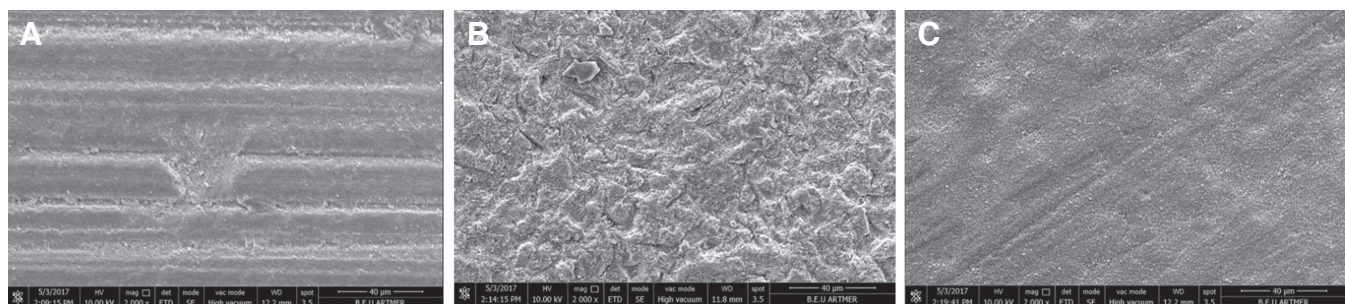
Surface treatment	Cnt		Tbc		Lsr	
RMC	Mean / SD	Differences*	Mean / SD	Differences*	Mean / SD	Differences*
FHC	14.15 (1.70)	Aa	16.41 (1.47)	Aa	19.62 (1.17)	Ba
RNC	14.78 (1.44)	Aa	15.52 (1.60)	Aa	19.69 (1.92)	Ba
PIHC	15.35 (1.62)	Aa	18.07 (2.89)	Ba	19.09 (2.30)	Ba

\*Statistical comparisons between hybrid ceramic/surface treatment groups were shown as letters and values having same letters are not significantly different for Tukey HSD test ( $P > .05$ ). \*\*The capital letters indicate the differences between the surface treatment groups and small caps indicates the differences between hybrid ceramic groups.

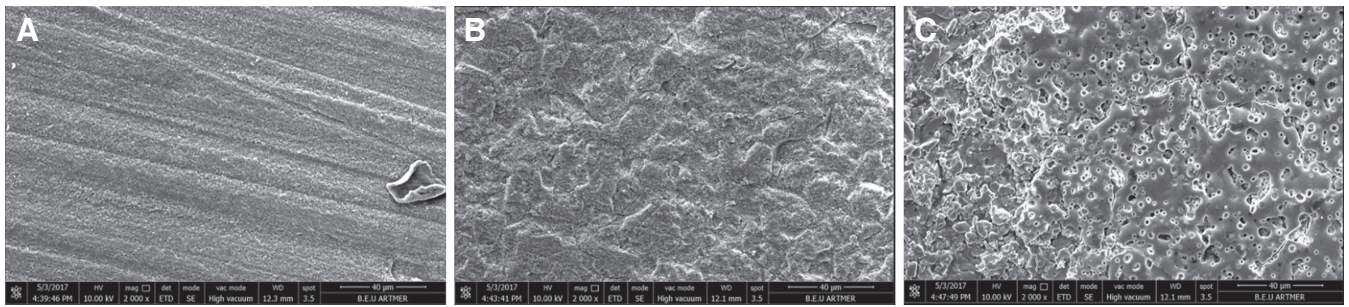
ence observed among the remaining groups ( $P > .05$ ).

The SEM images of test groups were shown in Fig. 2, Fig. 3, and Fig. 4. Prominent changes were observed on the topographical surface of all treated groups when compared to the control specimens. There were significant differences between the groups of RMC treated with the same surface treatment in the SEM images. While all RMCs surfaces after Tbc application had an irregular and rough appearance, it was seen that RNC specimens, in particular, contain micro-craters close to the sandblasted surface. For Lsr applied specimens, SEM images displayed irregular morphologic changes. Furthermore, in the PIHC group treated with Lsr, melted spots were seen on the surface of the material.

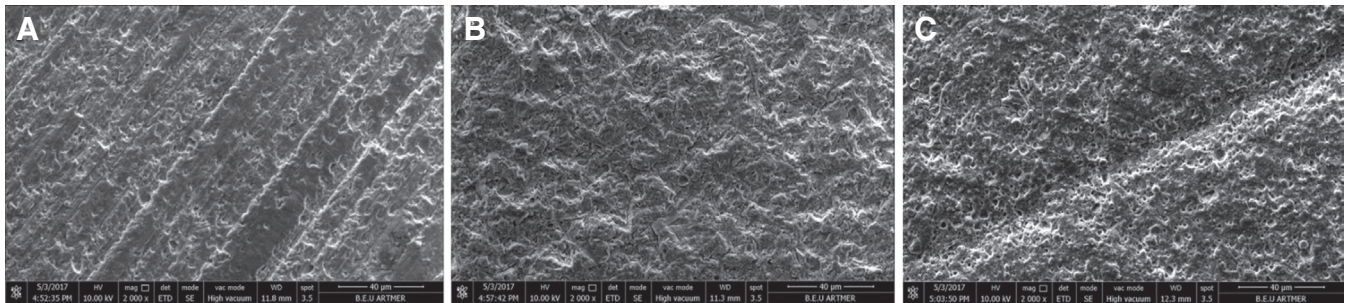
A statistically significant difference was determined among the test groups according to the Pearson Chi-Square test ( $P < .001$ ). When the fracture modes were analyzed, predominately cohesive or mixed type fractures were observed for all groups. While the cohesive type of fracture was mainly concentrated for each surface treatment-applied RMC groups, cohesive type of fracture was observed for all specimens of Tbc-applied FHC and Lsr-applied all RMC groups (Table 5). As a result of Kendall's tau\_b correlation analyses, the coefficient of correlation between SBS and Fracture-Modes was statistically significant ( $P < .001$ ,  $r^2 = 0.532$ ), indicating that these 2 variables were moderately correlated.



**Fig. 2.** SEM images ( $\times 2000$  magnification) of FHC RMC groups: (A) Cnt, (B) Tbc, (C) Lsr.



**Fig. 3.** SEM images ( $\times 2000$  magnification) of PIHC RMC groups: (A) Cnt, (B) Tbc, (C) Lsr.



**Fig. 4.** SEM images ( $\times 2000$  magnification) of RNC RMC groups: (A) Cnt, (B) Tbc, (C) Lsr.

**Table 5.** Summary of the fracture modes results of test groups

RMC	Surface treatment	Fracture type		
		Adhesive	Mixed	Cohesive
FHC	Cnt	3	1	6
	Tbc	0	0	10
	Lsr	0	0	10
RNC	Cnt	3	6	1
	Tbc	4	0	6
	Lsr	0	0	10
PIHC	Cnt	2	1	7
	Tbc	3	0	7
	Lsr	0	0	10

## DISCUSSION

The present study evaluated the effect of different surface treatments on the SBS of an IRC material on various RMC blocks. According to the study results, the first null hypothesis that the different surface treatment applications would not affect the SBS of an IRC to RMC blocks was rejected. While the SBS values were not varied depending on the RMC materials, the second null hypothesis accepted.

The microstructure of CAD/CAM ceramic blocks influences the aesthetic properties. There are critical relations among chemical composition, fabrication process, microstructure, and aesthetic properties of CAD/CAM ceramics.<sup>9,17,18</sup> The RMC materials have a composite structure that consists of both organic matrix and highly filled ceramic particles. This complex structure may provide a superior feature that allows a high degree of characterization extraorally or intraorally.<sup>4,17-20,34</sup> The aesthetic outcome of CAD/CAM made RMC restorations will be questionable in the anterior region. The veneering and characterization techniques will be suitable to overcome these aesthetic problems.<sup>2,23-30</sup> In the present study, the effectiveness of additional surface treatment application on the bonding performance of veneering IRC to RMC was investigated.

Until today, a wide variety of surface treatments have been used in the studies related to the SBS of resin composites to ceramic materials.<sup>2,29,30</sup> In most of these studies, it has been reported that the treatment of the ceramic surface with HF acid application followed by silane coupling agent was obtained a high bond strength.<sup>35</sup> Chemical etching with HF acid selectively dissolves the glassy matrix of ceramic, thus creating surface microporosities and irregularities to allow the infiltration of silane coupling agent for providing micromechanical retention and chemical bonding ability with ceramic and the overlaying material.<sup>22,31</sup> As well as positive effects of HF acid on the SBS of ceramic materials, in a study that was comparing the success of lithium disilicate, feldspathic ceramic and RNC, the researchers found that

the application of HF acid to RNC could alter the surface characterization and reduce the bond strength of the materials.<sup>22</sup> This results may be due to the fact that the application of HF acid causes the fillers in the inorganic structure of the ceramics to detach from the material structure and thus adversely affects the bond strength.<sup>22</sup> Also, HF acid is toxic and caustic, and represents a potential health hazard due to its toxicity and volatility.<sup>15,35</sup> Therefore, the questionable effects of HF acid on the bond strength of RMC, the acid roughening surface treatment protocol was not used in our study.

Airborne particle abrasion is another technique for creating a roughened surface on ceramics in prosthodontic laboratories or dental clinics. However, it can be claimed that the airborne particle abrasion surface treatment technique does not increase the micromechanical interlocking.<sup>15,35</sup> Kern and Thompson<sup>36</sup> have found that sandblasting can damage material surfaces and cause a large volume loss. This result was in agreement with another study revealing that sandblasting can damage material surfaces, partially destroy the resin matrix, and expose filler particles in resin-based materials.<sup>11</sup> Baruticigil *et al.*<sup>11</sup> also pointed out that it may be necessary to avoid blasting ceramic restorations. Tbc technique is more suitable than airborne particle abrasion for surface treatment.<sup>36</sup> Tbc or silicatization using CoJet in dentistry is a commonly used successful surface treatment method for ceramic materials. This is a cold silicatization method, and this method transfers the required kinetic energy to the object material surface for the silicatization process and does not require any additional heat or light application.<sup>36,37</sup> In addition, the CoJet system is based on airborne abrasion with especially silica-modified aluminum trioxide micro-blasting sand. The specific micro-blasting sand provides a reactive silica-rich treated surface. This surface structure makes the treated area suitable for the adhesion and silanization of the veneer resin material.<sup>37</sup> For these reasons in the present study, instead of airborne particle abrasion application, Tbc techniques with CoJet system was preferred as one of the surface treatment processes.

In the present study, it has been determined that the most effective surface treatment process for PIHC is Tbc surface treatment technique than the other RMCs. The most probable reason of this result is that the Tbc method has different effects on the bond strength of the RMCs due to the material contents. Since sand particles cannot reach the same depth in each material, the bond strength may be low on surfaces with limited sanding efficiency. In addition, the bond strength may be reduced because silicatization will be limited on surfaces where Tbc activity is limited.<sup>32</sup> On the other hand, PIHC material comprises a dominant ceramic network (75 vol%, 86 wt%) strengthened by an acrylic resin polymer network (25 vol%, 14 wt%) higher than other RMC materials.<sup>16</sup> So, the effectiveness of the Tbc method may have been greater on PIHC material than other RMCs.<sup>4</sup> This method can significantly increase the bond strength between PIHC surfaces and IRC by increasing the silica content on the ceramic surface.<sup>12,25</sup>

However, Tekçe *et al.*<sup>38</sup> found that the increased inorganic content of the PIHC material could lead to the formation of brittle structure than FHC. In that study, after 5000 thermal cycling, bond strength of CAD/CAM slabs from the same CAD/CAM PIHC block material using dual-cure adhesive cement for 60 s to sandblasted specimens of PIHC ceramics (22.8 MPa) was significantly lower than that for 15 s (33 MPa) and also that for 30 s (35.3 MPa) specimens ( $P < .001$ ). According to these results, increasing the ceramic composition of the materials makes them undented to the creation and propagation of cracks, and this may impair the mechanical behavior of restorative materials.<sup>38</sup> The results obtained by Tekçe *et al.*<sup>38</sup> and our study have varied, probably due to the difference in the veneering material, adhesive material, sandblasting time, and thermal cycling procedures.

There are some critical parameters in Tbc methods: nozzle distance from the substrate surface, impact angle to the substrate surface, working time, coverage area, and operating air pressure.<sup>36</sup> But generally, the manufacturers may not provide information about these parameters for sandblasting of the CAD/CAM materials that should be considered in the Tbc method. Working time varies throughout the literature.<sup>11,12,14</sup> These different working time can influence the creation of surface irregularities/micro-cracks in CAD/CAM RMC blocks, and can lead to varying degrees of roughness on blocks. Therefore, Tbc application for CAD/CAM RMCs should be specific to the material.<sup>14</sup> In our study, PIHC samples were found to be more successful in terms of bond strength in all Tbc applied materials. This result may be due to the fact that the parameters (50- $\mu$ m silica-coated  $Al_2O_3$  airborne particles, for 10 second at 2 bar pressure from 10 mm distance of substrate surface) selected for the Tbc method are more suitable for PIHC. As in many studies,<sup>35,36</sup> the SBS test results for FHC and RNC specimens treated with Tbc surface treatment showed a significant increase compared to the Cnt group. This success of Tbc surface treatment is thought to result from the combination of chemical roughness provided by silica and mechanical roughness provided by  $Al_2O_3$ .<sup>36</sup>

Er:YAG (erbium: yttrium, aluminum, garnet), Nd:YAG, and Er,Cr:YSGG (erbium, chromium: yttrium, scandium, gallium, garnet) lasers have been considered as an alternative surface treatment to condition the surfaces of dental materials.<sup>39</sup> The laser systems work with similar mechanism and they have the capability to remove particles from surfaces by micro explosion and vaporization, a process named ablation.<sup>39,40</sup> With this procedure, the micromechanical retention increases.<sup>31</sup> The wavelength of Er:YAG (2940 nm), Er,Cr:YSGG (2780 nm) and Nd:YAG (1064 nm) lasers are different from each other<sup>39,40</sup> and these differences may lead to different surface characterization of similar materials. Kamel *et al.*<sup>41</sup> used the laser application with 2 W power, 2780 nm wave length, 20 Hz repetition rate for 20 seconds at 1 mm distance from the surface of the PIHC specimens to compare the effect of the Er,Cr:YSGG and HF acid surface treatments. According to the results of this study, it

was observed that the interaction of Er,Cr:YSGG pulsed laser with the surfaces of CAD/CAM ceramic materials tested varies and this difference is related to the crystal structures of the materials used.<sup>41</sup> In our study, Nd:YAG laser application (3 W power, 1064 nm wave length, 20 Hz repetition rate for 20 seconds at 10 mm distance) was used for this aim. No matter the laser type and usage characteristics, in both studies, on irradiated surfaces of laser-applied PIHC samples, there was evidence of ablation and melting of the surface. Further, this surface characterization was not favorable for bonding.<sup>41</sup> Therefore, in our study, the Nd:YAG laser irradiation actually produced the lower SBS values for the PIHC specimens than other RMC materials. Erosion and melting were observed on the laser-irradiated PIHC surface. Fissures and cracked areas were not encountered. This result may have been due to the local temperature change caused by the non-cooled Nd: YAG laser process. Also, the non-cooled Nd:YAG laser treatment may have caused internal tensions that could damage the PIHC materials. The use of lower power of Nd:YAG laser with constant water cooling may reduce thermal side effects on PIHC materials.<sup>36</sup> Cengiz-Yanardag *et al.*<sup>42</sup> compared the effect of 2 W and 3 W Er,Cr: YSGG laser surface treatment (a repetition rate of 10 Hz, and 140  $\mu$ s pulse duration with 55% water and 65% air for 20 sec.) on RMCs. According to the results of this study, RNCs and FHCs 2W Er,Cr:YSGG laser treated groups showed significantly lower SBS values while there was no significant difference among control, 3W Er,Cr:YSGG laser and sandblasting groups. In the same study, for RNCs 3W Er,Cr:YSGG laser group showed lower bond strength values compared to control and 2W laser groups. According to Cengiz-Yanardag *et al.*,<sup>42</sup> the low bond strengths resulting from laser surface treatment may be due to thermal surface damage caused by laser power settings. In contrast; RNCs and FHCs evaluated in our study, 3W Nd:YAG laser applied groups showed higher bond strength values compared to Cnt groups. In short, Nd: YAG lasers can be a good alternative to the surface treatment of RMCs if the most appropriate parameters are selected for each material.

The SEM images also supported the results of these SBS values by the prominent changes determined on the topographical surface of Lsr and Tbc applied RMC materials, compared to Cnt groups (Fig. 2, Fig. 3, and Fig. 4). In the SEM images, it was observed that the Lsr applied surfaces had a large number of defects and retentive area. These findings will be interpreted that the Lsr treated surfaces are able to provide more retentive surface than Cnt groups. In other word, these results indicated that Lsr surface treatment increases the initial bond strength between the IRC and RMC.

Surface treatment methods lead to partially destroying the resin matrix and exposing filler particles to resin-based materials such as IRC.<sup>27</sup> Silanization is indispensable to provide a resistant bonding after the above-mentioned surface treatment options. Thus, a better mechanical interlocking between treated surface and substrate may be achieved.<sup>28</sup>

The present study supported this idea, and it has been showed silane coupling agent application to the treated ceramic surface results in significant bonding between the IRC and RMC materials. Both the Lsr and Tbc specimens showed superior initial as well as durable SBS compared to the Cnt groups. The possible reason for this finding is that a strong chemical bonding is formed between the silane and the silica in the treated ceramic surface by means of a siloxane network.<sup>25</sup> For our study, this reasoning explains the higher prevalence of cohesive failures, especially in the Lsr treated ceramic specimens.

In a study that tested similar materials with the  $\mu$ SBS test method, Cengiz-Yanardag *et al.*<sup>42</sup> reported that the most common type of fracture in Lsr and Tbc treated specimens were adhesive fracture. But, in the current study, there were no adhesive - mix failures in Lsr and mix failure in Tbc groups. These findings mean that the bond strength of the IRC to ceramics exceeds the shear bond strength between the ceramic and the IRC. Also, these findings demonstrated that ceramic surface treatment with Lsr or Tbc is an effective method to improve SBS to RMC. The observation of cohesive failure may lead to the conclusion that the adhesion of IRC to the ceramic, regardless of its surface treatment, is sufficient within the limits of this study.

There is no definitive threshold bond strength value of veneering materials to RMC that can be regarded “clinically acceptable”. However, a clinically acceptable threshold of bond strength ranges for resin to ceramics was reported between 10 and 13 MPa.<sup>15,43</sup> In the present study, the bond strengths of all specimens were 17 MPa in average, so veneering of RMC with IRC seemed clinically acceptable method for ceramic characterization.

Nevertheless, the occurrence of cohesive failures can also be attributed to the bond test employed. Many studies that used SBS tests observed that this methodology often produces fracture away from the adhesion zone and may increase the likelihood of cohesive or combined fractures. Moreover, the incidence of cohesive fractures may be increased due to the nonhomogeneous stress distribution along the bonded interfaces in the SBS test procedure.<sup>39,44</sup> This nonhomogeneous interface stress distribution causes fractures to start from defective areas in the interface or material that exhibit high-stress concentration. Therefore, in the present study, regardless of the surface treatments applied to the ceramic, the type of fracture was mostly cohesive.

The design of this *in vitro* study has several limitations, so the results of this study should be carefully evaluated. One of the limitation of this study is that the SBS test has been widely used for *in vitro* investigations of resin ceramics, but it has some limitations about the uniform stress distribution across the material.<sup>4,15,35</sup> Another limitation is the simplified specimen design. This design allows for some basic bonding assessments under standardized and controlled conditions, but cannot simulate the complex interactions between the three-dimensionally shaped frameworks and veneering materials. The bond strength of different

types of IRC materials to RMC should be evaluated in further *in vitro* and *in vivo* studies using various surface treatments, different Nd:YAG laser irradiation time and power settings, different adhesive materials, and different adhesive systems. Also, the validity of using IRC as veneering or characterization materials to RMC should be confirmed in randomized clinical research.

## CONCLUSION

Within the limitations of this *in vitro* study, the following conclusions could be drawn the type of surface treatment, especially Lsr treatment, was significant on the SBS of IRC to the RMC blocks. Using Nd:YAG laser treatment significantly improved the SBS values for all of the RMC materials. In the case of the PIHC material, the tribochemical silica coating technique significantly improved the SBS values of the material compared to laser treatment. For promoting a reliable bond during characterization of ceramic restorations, Nd: YAG laser or tribochemical silica coating technique should be used, putting in consideration the microstructure and composition of RMC materials and appropriate parameters for each material.

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