# Effects of ytterbium laser surface treatment on the bonding of two resin cements to zirconia

Keita TOYODA<sup>1</sup>, Yusuke TANIGUCHI<sup>1</sup>, Keiko NAKAMURA<sup>2</sup>, Kota ISSHI<sup>3</sup>, Kae KAKURA<sup>1</sup>, Hiroshi IKEDA<sup>4</sup>, Hiroshi SHIMIZU<sup>4</sup>, Hirofumi KIDO<sup>1</sup> and Tomohiro KAWAGUCHI<sup>5</sup>

<sup>1</sup> Division of Oral Implantology, Department of Oral Rehabilitation, Fukuoka Dental College, 2-15-1 Tamura, Sawara, Fukuoka 814-0193, Japan

<sup>2</sup> Fukuoka Dental College Center for Oral Diseases, 3-2-1 Hakataekimae, Hakata, Fukuoka 812-0011, Japan

<sup>3</sup> Central Dental Laboratory, Fukuoka Dental College Hospital, 2-15-1 Tamura, Sawara, Fukuoka 814-0193, Japan

<sup>4</sup> Division of Biomaterials, Department of Oral Functions, Kyushu Dental University, 2-6-1 Manazuru, Kokura-kita, Kitakyushu, Fukuoka 803-8580, Japan

<sup>5</sup> Division of Removable Prosthodontics, Department of Oral Rehabilitation, Fukuoka Dental College, 2-15-1 Tamura, Sawara, Fukuoka 814-0193, Japan

Corresponding author, Tomohiro KAWAGUCHI; E-mail: tomokawa@college.fdcnet.ac.jp

Monolithic zirconia crowns bonded to zirconia abutments have become more commonly used in the construction of cement-retained implant superstructures. The present study aimed to examine the effects of laser surface treatments on the bond strength of two resin cements to zirconia. Three types of surfaces were examined: untreated, alumina blasted, and ytterbium laser treated; and two types of resin cements: 4-META/MMA-TBB resin cement and composite resin cement. Half of the specimens were subjected to a thermocycling process. Subsequently, a shear bond test was carried out. In addition, surface roughness was measured for each surface type. The results showed that laser treatment increased zirconia surface roughness and that laser treatment significantly increased shear bond strength after the thermocycling of both cement types compared to no treatment. Our experimental results suggested that ytterbium laser surface treatment of zirconia increased the bond strength of resin cements.

Keywords: Bond strength, Resin cement, Zirconia, Laser, Dental implant

# INTRODUCTION

Metals, such as titanium alloys, and ceramics have conventionally been used in superstructures and abutments in implant treatments for missing teeth. With the implementation of digital technologies such as dental CAD-CAM in recent years, yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) with high mechanical strength has been increasingly used in abutments and superstructures<sup>1)</sup>. Cement-retained superstructures have been constructed using titanium or zirconia abutments bonded to monolithic zirconia crowns with resin cement<sup>2)</sup>. Since there is no periodontal ligament around a zirconia restoration supported by an osseointegrated implant, occlusal force is more directly applied to such a structure compared to a natural tooth. Therefore, a stable bond is needed between zirconia and titanium or between two zirconia surfaces since detached zirconia crowns continue to be reported in clinical practice<sup>3,4)</sup>.

Several surface treatments, such as alumina blasting, the application of zirconia priming agents containing functional monomers, the laser treatments, have been proposed to improve the bond strength to zirconia<sup>5-13)</sup>. Alumina blasting with alumina particles has been considered an effective zirconia surface treatment<sup>5-8)</sup>. Alumina blasting can improve bonding by micromechanically roughening a zirconia surface in the laboratory, and furthermore it can remove saliva contaminants that inhibit adhesion after try-in prostheses on the chair side<sup>14,15)</sup>. Another effective treatment uses an acid functional monomer, particularly 10methacryloyloxydecyl dihydrogen phosphate (MDP)<sup>9-11)</sup>. The use of priming agents containing MDP has been encouraging for enhancing the bond strength of resin cements on zirconia<sup>16,17)</sup>. Laser irradiation (CO<sub>2</sub> and YAG) is another method for roughening the zirconia surface<sup>18,19)</sup>. Recent studies have examined laser surface treatment to improve the surface modification of titanium and zirconia implant bodies<sup>12,13)</sup>. Laser treatment can cause various changes in material surfaces, including creating proper surface roughness to increase the bonding of resin cements to zirconia, which is difficult to manufacture with high hardness<sup>18-24)</sup>. It has been also recognized that ytterbium laser treatment on zirconia surfaces enhances the bond strength between zirconia and resin cements<sup>23)</sup>.

The currently available resin cements can be categorized into two types: (1) composite type resin cements consisting of inorganic fillers embedded in an organic matrix and (2) methyl methacrylate (MMA)type resin cements composed of a liquid, containing a functional monomer (4-META; 4-methacryloxyethyl trimellitate anhydride), MMA and an initiator (TBB; tri-n-butylborane) and a polymethyl methacrylate (PMMA) powder<sup>15,25</sup>. Composite type resin cements are

Color figures can be viewed in the online issue, which is available at J-STAGE.

Received Jan 28, 2021: Accepted Jun 21, 2021

doi:10.4012/dmj.2021-036 JOI JST.JSTAGE/dmj/2021-036

the standard material for the cementation of implant prosthesis<sup>2,26</sup>. Conventional MMA-type resin cement has also been generally used as a resin cement for bonding to tooth structures<sup>27.30</sup>.

No study, however, has been reported on ytterbium laser treatment to improve the bonding of both types of resin cements to zirconia. Only a few reports have examined how bond strength is affected by differences in alumina blasting treatment and laser treatment<sup>23</sup>). Therefore, our study examined how alumina blast and ytterbium laser treatments on zirconia surfaces affected the bond strength of two resin cements. The null hypothesis was that there would be no difference in the shear bond strength to zirconia between the surface preparations with two resin cements.

## MATERIALS AND METHODS

#### Shear bond test

Table 1 shows the materials used in our study. Adherends were made using zirconia powder (HSY-3 FSD, Daiichi Kigenso Kagaku Kogyo, Tokyo, Japan) and a cold isostatic press (DR. CIP-M, Kobe Steel, Hyogo, Japan) followed by sintering at 1,500°C. The process produced 150 zirconia plates (yttria-stabilized tetragonal zirconia polycrystal ceramics). The final specimens were each a 10-mm square with 1-mm thickness.

For each zirconia plate, the surface to be bonded was polished with 600 grit waterproof sandpaper

(Waterproof paper, Riken Corundum, Saitama, Japan) under running water. The plates were divided into 3 groups by surface treatment method: untreated, alumina blasted, and laser treated groups. The untreated group had untreated surfaces after polishing. In the alumina blasted group, the surfaces were treated using a sandblaster (DUOSTAR Z2, BEGO, Bremen, Germany). Its nozzle was positioned perpendicularly to the surface to be bonded and at a distance of approximately 10 mm from the surface. Alumina particles with a 50-µm diameter were blasted at a pressure of 0.3 MPa. In the laser-treated group, the surfaces were irradiated in a fixed direction using a ytterbium fiber laser system (MD-F3000, Kevence, Osaka, Japan) at a power output of 24 W and pulse frequency of 60 kHz for 6.6 s of laser irradiation time.

Two types of adhesive materials were used: 4-META/ MMA-TBB resin cement (Super-Bond C&B, Sun Medical, Shiga, Japan) and composite resin cement (Panavia V5, Kuraray Noritake Dental, Tokyo, Japan). The primers were PZ PRIMER (Sun Medical) for the former cement and Clearfil Ceramic Primer Plus (Kuraray Noritake Dental) for the latter cement.

A uniform bonding surface area was established among the specimens by placing masking tape with a 6-mm diameter hole on each surface to be bonded and then placing a Teflon tube (5-mm inner diameter, 6-mm outer diameter and 5-mm length) into the hole. Primer was applied onto the surface with a sponge brush according

Material/Trade name	Manufacturer	Components
Zirconia powder		
HSY-3FSD	Daiichi Kigenso Kagaku Kogyo	97% ZrO <sub>2</sub> , 3% Y <sub>2</sub> O <sub>3</sub>
4-META/MMA-TBB res	in cement	
Super-Bond C&B	Sun Medical	Powder: PMMA, TiO <sub>2</sub> Liquid: 4-META, MMA, Catalyst: TBB
Composite resin cement	t	
Panavia V5	Kuraray Noritake Dental	Bis-GMA, TEGDMA, Silanated barium glass filler, Silanated fluoroalminosilicate glass filler, Colloidal silica, Surface treated aluminum oxide filler, Hydrophobic aromatic dimethacrylate, Hydrophilic aliphatic dimethacrylate, dl-Camphorquinone, Initiators, Accelerators, Pigments
Priming agent		
PZ Primer	Sun Medical	Liquid A: MMA, MDP, others Liquid B: MMA, 3-TMSPMA
Clearfil Ceramic Primer Plus	Kuraray Noritake Dental	MDP, 3-TMSPMA, ethanol

Table 1 Materials used in this study

PMMA: polymethyl methacrylate, 4-META: 4-methacryloxyethyl trimellitate anhydride, MMA: methyl methacrylate, TBB: Tri-n-butylborane, Bis-GMA: bisphenol A diglycidyl methacrylate, TEGDMA: triethyleneglycol dimethacrylate, hydrophobic MDP: 10-methacryloyloxydecyl dihydrogen phosphate, 3-TMSPMA: 3-(trimethoxysilyl) propyl methacrylate to the manufacturer's instructions and air dried. The Teflon tube was filled with an adhesive material while avoiding air bubbles. 4-META/MMA-TBB cement was applied inside the Teflon tube on each specimen using a micro syringe (Super-Bond micro syringe, Sun Medical) and allowed to stand for 1 h at room temperature after the tube was filled. The composite cement was cured for 10 s from one direction in normal mode (1,200 mW/cm<sup>2</sup>) using an LED curing unit (G-light Prima, GC, Tokyo, Japan). Each specimen was immersed in 37°C purified water for 24 h (Pure water, Wako, Osaka, Japan). The Teflon tube and masking tape were removed, and the specimen was subjected to a shear bond test (Fig. 1).

All specimens were then divided into either a group that was subjected to 10,000 thermocycles (TC10,000 group) or a group not subjected to a thermocycle (TC0 group). Each thermocycle consisted of alternating water immersion at 5°C for 1 min and 55°C for 1 min.

The specimens underwent a shear bond test using a universal testing device (Autograph, AGS-J, Shimadzu,



Fig. 1 Diagram of specimen.



Fig. 2 Diagram of the specimen on the mechanical testing machine.

Kyoto, Japan) whose blade-shaped loading device applied a load parallel to the bonding surface at a crosshead speed of 0.5 mm/min, and the shear bond strength was measured. Shear bond strength (MPa) was defined as the maximum load required to fracture divided by the bonded surface area (Fig. 2). There were 12 specimens each in the TC0 group and the TC10,000 group.

*Examination of surface properties after surface treatment* The surface properties of untreated, alumina blasted, and laser-treated surfaces were examined using scanning electron microscopy (SEM; JSM-6330F, JEOL, Tokyo, Japan) and laser scanning microscope (VK-X100, Keyence). SEM was used at an accelerating voltage of 5 kV and 500× magnification.

Measurement of surface roughness after surface treatment A laser scanning microscope was used to measure  $50 \times 50$  µm areas. The surface roughness was calculated as the arithmetic mean height of the surface (Sa) and developed interfacial area ratio (Sdr). Each value was calculated as the mean value of three randomly selected sites on a specimen surface. In addition, the sample surfaces were observed with a scanning area of  $200 \times 282.9$  µm after applying each surface treatment.

# Examination of the fracture surface after the shear bond test

For each specimen, the fractured surface after shear bond testing was examined using a SEM under the same conditions as those in the surface property examination. The failure mode was divided into three types: adhesive failure between cement and zirconia, cohesive failure of cement, and mixed failure with residual cement in some areas of the fracture surface.

#### Statistical analysis

The results of the shear bond test were processed using statistical analysis software (IBM SPSS ver.18, IBM Japan, Tokyo, Japan). All data were analyzed with the Kolmogorov-Smirnov test to evaluate the normality, with the Levene test to evaluate the homoscedasticity. The shear bond strength data were analyzed with analysis of variance (ANOVA) for each resin cement followed by Bonferroni's multiple comparison test at the level of statistical significance ( $\alpha$ =0.05).

# RESULTS

# Shear bond test

The results of two-way ANOVA for each resin cement (Tables 2 and 3) showed that there were significant differences between the surface preparation (p<0.001) and thermocycling condition (p<0.001). There was also a significant interaction between the surface preparation and thermocycling condition (p<0.001) for shear bond strengths. Therefore, the bond strengths were analyzed by one-way ANOVA and Bonferroni's multiple comparison test. Figure 3 shows the results of the shear bond strength of 4-META/MMA-TBB resin cement and

Source	Sum of squares	Degrees of freedom	Mean squares	$\mathbf{F}$	<i>p</i> value
Surface preparation	1,316.462	2	658.231	91.867	0.000
Thermocycling condition	922.082	1	922.082	128.692	0.000
Surface preparation* Thermocycling condition	439.578	2	219.789	30.675	0.000
Error	472.892	66	7.165	_	—
Total	20,928.973	72	_	—	

Table 2 Results of two-way ANOVA for shear bond strength data of 4-META/MMA-TBB resin cement with surface preparation and thermocycling condition

 Table 3
 Results of two-way ANOVA for shear bond strength data of composite resin cement with surface preparation and thermocycling condition

Source	Sum of squares	Degrees of freedom	Mean squares	F	p value
Surface preparation	116.798	2	58.399	7.527	0.001
Thermocycling condition	1,845.636	1	1,845.636	237.889	0.000
Surface preparation* Thermocycling condition	112.083	2	56.042	7.223	0.001
Error	512.053	66	7.758		
Total	6,595.920	72	_	_	_





the statistical analysis results. The mean shear bond strength before thermocycling ranged from 9.8 to 14.5 MPa. The data were analyzed as having normality and homoscedasticity in the bond strength. The shear bond strength of 4-META/MMA-TBB cement was lowest for no treatment group, and the laser treatment and alumina blast treatment groups had significantly higher shear bond strength than no treatment group (p<0.05). The mean shear bond strength after thermocycling ranged from 10.0 to 25.5 MPa. The shear bond strength of 4-META/MMA-TBB cement was lowest for no treatment group, and there was no significant difference between



Fig. 4 Results of shear bond strength of composite resin cement (MPa). Categories with the same letter were not significantly different (p>0.05).

alumina blast treatment and laser treatment (p>0.05). When the shear bond strength was compared before thermocycling and after thermocycling, the no treatment groups showed no significant difference (p>0.05). The laser treatment and alumina blast treatment groups, however, showed a significant difference in bond strength before thermocycling and after thermocycling (p<0.05).

Figure 4 shows the results of the shear bond strength of composite resin cement and the statistical analysis results. The mean shear bond strength before thermocycling ranged from 11.2 to 14.4 MPa. There was no significant difference in the shear bond strength of the

cement among each treatment (p>0.05). The mean shear bond strength after thermocycling ranged from 0.1 to 5.2 MPa. Application of laser treatment yield significantly higher bond strength than no treatment (p<0.05), and there was no significant difference between alumina blast treatment and no treatment (p>0.05). When the shear bond strength was compared before thermocycling and after thermocycling, there were significant differences for no treatment, alumina blast treatment, and laser treatment groups (p<0.05).

#### Examination of fracture surface

Tables 4 and 5 show the results of the failure modes after the shear bond test. In the pre-thermocycled groups, the most common failure mode was mixed failure for 4-META/MMA-TBB resin cement. There was cohesive failure in one specimen in each of the alumina blast treatment group and laser treatment group. In the postthermocycle groups, there was no cohesive failure, and the most common failure mode was adhesive failure. There were also some specimens with mixed failure. When the pre-thermocycled groups were examined for the composite resin cement, some no treatment specimens had mixed failure, but all alumina blast treatment and laser treatment specimens had adhesive failure. In the post-thermocycled groups, the fracture mode was adhesive failure in all no treatment, alumina blast, and laser treatment specimens.

Figure 5 shows SEM images of typical fracture surfaces after the shear bond test for 4-META/MMA-

#### Table 4 Failure modes of 4-META/MMA-TBB resin cement after shear bond test

		TC0			TC10,000	
	Cohesive	Mixed	Adhesive	Cohesive	Mixed	Adhesive
No treatment	0	11	1	0	2	10
Alumina blast treatment	1	11	0	0	2	10
Laser treatment	1	11	0	0	4	8

#### Table 5 Failure modes of composite resin cement after shear bond test

	TC0			TC10,000		
	Cohesive	Mixed	Adhesive	Cohesive	Mixed	Adhesive
No treatment	0	4	8	0	0	12
Alumina blast treatment	0	0	12	0	0	12
Laser treatment	0	0	12	0	0	12





Fig. 5 SEM images of zirconia surface and 4-META/ MMA-TBB resin cement before and after thermal cycling (×500): (A) no treatment, (B) alumina blast treatment, and (C) laser treatment.

Arrows in SEM indicate residual resin cements (bar=30  $\mu m$ ).

Before thermal cycling



Fig. 6 SEM images of zirconia surface and composite resin cement before and after thermal cycling (×500): (A) no treatment, (B) alumina blast treatment, and (C) laser treatment.

B

Arrows in SEM indicate residual resin cements (bar= $30 \ \mu m$ ).

TBB resin cement. Arrows on the images show residual resin cement on the surfaces. On the laser-treated surface after thermocycling, resin cement adhered to the laser markings. Figure 6 shows SEM images of typical fracture surfaces after the shear bond test for composite resin cement. In the images after thermocycling, only the laser-treated surface showed residual resin cement.

*Examination of surface properties after surface treatment* Surface properties after surface treatment were examined by SEM. The untreated surface was flat and showed slight scratches from polishing with waterproof sandpaper. The alumina blasted surface showed fine unevenness. The laser-treated surface showed laser markings whose grooves were formed parallel to each other with a uniform spacing between them (Fig. 7).

Measurement of surface roughness after surface treatment Figure 8 shows laser scanning microcopy images of untreated, alumina blasted, and laser-treated zirconia surfaces. The arithmetic mean height (Sa) was  $0.19\pm0.06$ ,  $0.55\pm0.05$ , and  $2.39\pm0.02$  µm for untreated, alumina blasted, and laser-treated surfaces, respectively (Table 6). The developed interfacial area ratios (Sdr) were  $0.11\pm0.05$ ,  $1.85\pm0.12$ , and  $3.11\pm0.39$ .



Fig. 7 SEM images of surfaces after surface treatment (×500): (A) no treatment, (B) alumina blast treatment, (C) laser treatment (bar=30 μm).

# DISCUSSION

Various surface treatments of zirconia have been studied to improve its bonding with teeth and metals, and bond strength has been reported to be affected by the pretreatment of zirconia surfaces<sup>3,25,31)</sup>. For metallic restorations, bond strength is known to be improved by surface blasting which increases mechanical interlocking. It is also known to be improved by surface treatment with a primer, a monomer modified in sulfur for precious alloys and a primer containing a phosphate ester monomer or carboxylic acid monomer for nonprecious alloys<sup>32,33)</sup>. Zirconia, however, has a low potential for chemical bonding. As mentioned previously, zirconia has been reported to have issues of fracture and detachment at the bonded area in clinical reports and clinical outcomes<sup>3,4)</sup>. In recent years, there has been increasing use of zirconia in abutments and superstructures with the implementation of digital technology in implant therapy. Thus, there is a pressing need to improve adhesive technology for zirconia.

Many studies have been conducted to improve the bonding of zirconia. The two primers in our study contained both MDP and 3-TMSPMA. It was reported that the application of primers containing MDP was effective in improving the bond strength of composite cement to zirconia<sup>16,17</sup>. The phosphate ester group of MDP creates chemical bonds with metal oxides such as zirconia<sup>34</sup>. In addition, the amount of MDP also plays a critical role in bonding between cement and zirconia<sup>9,35</sup>. The difference in the bond strength between the two types of resin cements after thermal cycling in this study might be affected by the concentration of MDP and 3-TMSPMA contained in the primer.

Silanes such as 3-TMSPMA are commonly incorporated in dental adhesives containing MDP to expand their applications in ceramic bonding<sup>21,28)</sup>. However, traditional silane-coupling agent like



Fig. 8 Laser scanning microscopy images of the zirconia surface: (A) no treatment, (B) alumina blast treatment, (C) laser treatment.

Table 6 Surface roughness value of the zirconia surface after surface treatments

	No treatment	Alumina blast treatment	Laser treatment
Sa (µm)	$0.19 \pm 0.06$	$0.55 \pm 0.05$	$2.39 \pm 0.02$
Sdr	$0.11 \pm 0.05$	$1.85 \pm 0.12$	$3.11 \pm 0.39$

3-TMSPM is not effective with zirconia due to the lack of silica in zirconia. The unreacted residual monomer of 3-TMSPM might exist in primer layer. Therefore, it is assumed that the use of 3-TMSPM may interfere the chemical reaction of resin-zirconia bonding<sup>9,17)</sup>. It has also been reported that the combined application of silane and MDP is currently one of the most reliable bonding systems for zirconia<sup>25)</sup>. 4-META and MDP contained in resin cements also play a major part in the bonding of resin cement to zirconia<sup>15,25)</sup>. Previous studies have proved that 4-META and MDP function as coupling agents due to the chemical reaction between the hydroxyl groups in 4-META or the hydrogen groups in MDP and zirconia, similar to the reaction between silane coupling agents and silica-based ceramics<sup>28)</sup>. It was confirmed that MMA-type resin cement produces greater bond strength than other resin cements<sup>28-30</sup>. Micromechanical retention is essential for the bonding of resin cement. The higher bond strength in MMAtype resin cement could be attributed to its higher micromechanical retention formed due to its excellent flowability<sup>29</sup>. This study showed similar results.

In this study, the bond strength of 4-META/MMA-TBB resin cement of the alumina blast and laser treatment groups after thermal cycling increased significantly. Since the thermal cycling process is an acceleration test that influences the bond between cements and zirconia, the bond strength usually decreases due to the difference in their thermal expansion coefficients<sup>36)</sup>. In this study, the number of adhesive failures increased for both cements (Tables 4 and 5). However, the phenomenon where the bond strength increases after thermal cycling has been occasionally reported in a previous study using the 4-META/MMA-TBB resin cement<sup>27)</sup>. The discrepancy could be attributed to the difference in polymerization methods. In dual-cure composite cements, the initial polymerization can be effectively accelerated by light irradiation. On the other hand, since 4-META/MMA-TBB resin cement is a selfcuring resin cement, the speed of polymerization may not have sufficiently progressed to provide the initial bond strength before thermal cycling<sup>27)</sup>. Therefore, the polymerization of 4-META/MMA-TBB resin cement may have proceeded during the thermal cycling treatment, which was able to result in the increased bond strength observed. In addition, in this study, the shear bond strength values of the composite resin cement group after thermal cycling were totally lower than those in other previous studies<sup>17)</sup>. This is due to the thermal cycling conditions in this study. The test specimens for thermal cycling were performed with the Teflon tube surrounding the cement removed. Consequently, the thermal stress from thermal cycling could have increased, and the shear bond strength might be considered to be lower than that in other previous studies<sup>15,17)</sup>.

The modification of surface properties is an important factor in increasing bonding. Appropriate surface roughening increases the surface area and is effective in increasing bonding by interlocking. Okada *et al.* reported that zirconia surface roughness achieved

by alumina blast treatment ranges from 0.27 to 0.45 um at different pressures<sup>37)</sup>. Another study used a tribochemical reaction to increase the bonding of zirconia<sup>38,39</sup>. When the atomic layers at the surface of a material are subjected to friction, the bonds between atoms are dissociated, causing various reactions to occur at lower energies than conventional chemical reactions. When alumina particles are thinly coated with  $SiO_2$  and blasted, the zirconia surface is roughened, and strong bonds are formed between the zirconia surface and thin  $SiO_2$  film due to the energy of impact. This treatment has been reported to make silane coupling effective by simultaneously roughening the zirconia surface and forming a SiO<sub>2</sub> film<sup>40</sup>. However, Kern and Thompson reported that stable silicate layers cannot be formed by tribochemical treatment of high-strength, high-density ceramics<sup>41</sup>. Therefore, one cannot expect a substantial increase in bond strength using a tribochemical treatment of zirconia. Alumina blast treatment has long been used to roughen the surfaces of restorations for bonding.

Ruja et al. treated zirconia surfaces with an ultrashort-pulse laser and measured the bond strength between composite resin cement and the zirconia surfaces<sup>12)</sup>. They found that this bond strength after laser surface treatment was approximately 35% higher than that after alumina blasting. Typical laser sources used in surface treatment are a CO<sub>2</sub> laser, a gas laser using CO<sub>2</sub> as the medium, and a YAG laser, YVO laser, and solid-state laser using solids (such as those containing yttrium) as the medium. A  $CO_2$  laser is characterized by its high energy efficiency and helium, which enables a continuous, stable beam quality. Although this laser is used on a wide variety of materials, it tends to heat materials and is, therefore, not suitable for the treatment of metals. A YAG laser and YVO laser are used to treat metals and ceramics. A fiber laser is a solid-state system whose medium is optical fibers doped with the rare-earth element vtterbium (Yb). This laser has a very small focal point diameter and can treat metals and ceramics. It is more suitable for fine and deep treatment than YAG or YVO lasers.

Previous studies have investigated the ability of different laser types to enhance the bond strength of resin cements to zirconia<sup>18,19,24)</sup>. Akın et al. reported that CO<sub>2</sub> laser irradiation of zirconia might be an ineffective method for improving the bond strength of resin cement to zirconia<sup>19)</sup>. Er:YAG and Nd:YAG laser treatments have been proposed to improve the bond strength to zirconia<sup>19,22</sup>. In this study, the bond strength data proved that ytterbium fiber laser irradiation was more efficient at increasing the bond strength of resin cements to zirconia. This result agreed with the results of a previous study that reported the use of ytterbium fiber laser irradiation<sup>23)</sup>. Kakura *et al.* used a YAG laser for surface modification of zirconia implants<sup>42)</sup>. They achieved a surface roughness (Sa) of approximately 3.33  $\mu$ m, but the laser treatment caused small defects on the zirconia surface. Taniguchi et al. used a fiber laser to modify the zirconia implant surface and created surface structures with continuous grooves 50 µm wide and 20 um deep<sup>13)</sup>. A fiber laser can achieve a smaller focus diameter than other types of lasers and is thus, suitable for minute treatment. Our study used a fiber laser surface treatment following the methods of Taniguchi et al. We were able to achieve a surface roughness of approximately 2.39 µm with fiber laser treatment (Fig. 8). This surface roughness value produced the highest bond strength for laser treated surfaces. This result was thought to be due to increased surface area from surface roughening and due to micromechanical interlocking (Figs. 5 and 6). There is no concern of a contaminated bonding surface in laser treatment, unlike in alumina blasting, and laser treatment can be expected to be highly effective as a surface treatment to increase bond strength. The influence of laser irradiation on the surface characteristics of zirconia is also controversial<sup>20,23,24)</sup>. However, there are only a limited number of studies reporting on the chemical characteristics<sup>43,44)</sup>.

Our study examined the effects of zirconia surface treatment on bond durability using 4-META/MMA-TBB resin cement and composite resin cement. The results showed that bond strength and post-thermocycling bond durability with both two cements were increased by roughening the zirconia surface using ytterbium laser treatment compared with no treatment. Since the ytterbium fiber laser used in our study is suitable for fine treatment, various surface properties can be created depending on the restoration morphology and materials and on the properties of the adhesives.

# CONCLUSION

Within the limitations of this study, the application of ytterbium laser surface treatment on zirconia could improve the shear bond strength of both 4-META/ MMA-TBB resin cement and composite resin cement to zirconia.

#### REFERENCES

- Raigrodski AJ, Hillstead MB, Meng GK, Chung K-H. Survival and complications of zirconia-based fixed dental prostheses: a systematic review. J Prosthet Dent 2012; 107: 170-177.
- Mehl C, Zhang Q, Lehmann F, Kern M. Retention of zirconia on titanium in two-piece abutments with self-adhesive resin cements. J Prosthet Dent 2018; 120: 214-219.
- Tzanakakis EG, Tzoutzas IG, Koidis PT. Is there a potential for durable adhesion to zirconia restorations? A systematic review. J Prosthet Dent 2016; 115: 9-19.
- 4) Pjetursson BE, Thoma D, Jung R, Zwahlen M, Zembic A. A systematic review of the survival and complication rates of implant-supported fixed dental prostheses (FDPs) after a mean observation period of at least 5 years. Clin Oral Implants Res 2012; 23: 22-38.
- Sato H, Yamada K, Pezzotti G, Nawa M, Ban S. Mechanical properties of dental zirconia ceramics changed with sandblasting and heat treatment. Dent Mater J 2008; 27: 408-414.
- 6) Kosmac T, Oblak C, Jevnikar P, Funduk N, Marion L. The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic. Dent Mater 1999; 15: 426-433.

- He M, Zhang Z, Zheng D, Ding N, Liu Y. Effect of sandblasting on surface roughness of zirconia-based ceramics and shear bond strength of veneering porcelain. Dent Mater J 2014; 33: 778-785.
- 8) Caravaca CF, Flamant Q, Anglada M, Gremillard L, Chevalier J. Impact of sandblasting on the mechanical properties and aging resistance of alumina and zirconia based ceramics. J Eur Ceram 2018; 38: 915-925.
- 9) Koko M, Takagaki T, Abdou A, Inokoshi M, Ikeda M, Wada T, et al. Effects of the ratio of silane to 10-methacryloyloxydecyl dihydrogenphosphate (MDP) in primer on bonding performance of silica-based and zirconia ceramics. J Mech Behav Biomed Mater 2020; 112: 104026.
- Souza G, Hennig D, Aggarwal A, Tam LE. The use of MDP based materials for bonding to zirconia. J Prosthet Dent 2014; 112: 895-902.
- 11) Awliya W, Oden A, Yaman P, Dennison JB, Razzoog ME. Shear bond strength of a resin cement to densely sintered high-purity alumina with various surface conditions. Acta Odontol Scand 1998; 56: 9-13.
- 12) Ruja MA. Souza GM, Finer Y. Ultrashort-pulse laser as a surface treatment for bonding between zirconia and resin cement. Dent Mater 2019; 35: 1545-1556.
- 13) Taniguchi Y, Kakura K, Yamamoto K, Kido H, Yamazaki J. Accelerated osteogenic differentiation and bone formation on zirconia with surface grooves created with fiber laser irradiation. Clin Implant Dent Relat Res 2016; 18: 883-894.
- Yoshida K. Influence of cleaning methods on resin bonding to saliva-contaminated zirconia. J Esthet Restor Dent 2018; 30: 259-264.
- 15) Moon JE, Kim SH, Lee JB, Ha SR, Choi YS. The effect of preparation order on the crystal structure of yttria-stabilized tetragonal zirconia polycrystal and the shear bond strength of dental resin cements. Dent Mater 2011; 27: 651-663.
- 16) Valente F, Mavriqi L, Traini T. Effects of 10-MDP based primer on shear bond strength between zirconia and new experimental resin cement. Materials (Basel) 2020; 13: 235.
- 17) Yagawa S, Komine F, Fushiki R, Kubochi K, Kimura F, Matsumura H. Effect of priming agents on shear bond strengths of resin-based luting agents to a translucent zirconia material. J Prosthodont Res 2018; 62: 204-209.
- 18) Akyil MS, Uzun IH, Bayindir F. Bond strength of resin cement toyttrium-stabilized tetragonal zirconia ceramic treated with air abra-sion, silica coating, and laser irradiation. Photomed Laser Surg 2010; 28: 801-808.
- 19) Akın H, Ozkurt Z, Kırmalı O, Kazazoglu E, Ozdemir AK. Shear bondstrength of resin cement to zirconia ceramic after aluminum oxidesandblasting and various laser treatments. Photomed Laser Surg 2011; 29: 797-802.
- 20) Tanış MÇ, Akçaboy C. Effects of different surface treatment methods and MDP monomer on resin cementation of zirconia ceramics an in vitro study. J Lasers Med Sci 2015; 6: 174-181.
- 21) Turker N, Özarslan MM, Buyukkaplan US, Başar EK. Effect of different surface treatments applied to short zirconia and titanium abutments. Int J Oral Maxillofac Implants 2020; 35: 948-954.
- 22) Okutan Y, Kandemir B, Gundogdu Y, Kilic HS, Yucel MT. Combined application of femtosecond laser and air-abrasion protocols to monolithic zirconia at different sintering stages: Effects on surface roughness and resin bond strength. J Biomed Mater Res B Appl Biomater 2021; 109: 596-605.
- 23) Unal SM, Nigiz R, Polat ZS, Usumez A. Effect of ultrashort pulsed laser on bond strength of Y-TZP zirconia ceramic to tooth surfaces. Dent Mater J 2015; 34: 351-357.
- 24) da Silva BTF, Trevelin LT, Schroeter AC, Willers AE, Cesar PF, Matos AB. Effect of silica coating and laser treatment on the flexural strength, surface characteristics, and bond strength of a dental zirconia. Eur J Oral Sci 2021; 129:

e12754.

- 25) Miyazaki T, Nakamura T, Matsumura H, Ban S, Kobayashi T. Current status of zirconia restoration. J Prosthodont Res 2013; 57: 236-261.
- 26) Rohr N, Brunner S, Märtin S, Fischer J. Influence of cement type and ceramic primer on retention of polymer-infiltrated ceramic crowns to a one-piece zirconia implant. J Prosthet Dent 2018; 119: 138-145.
- 27) Shinagawa J, Inoue G, Nikaido T, Ikeda M, Burrow MF, Tagami J. Early bond strengths of 4-META/MMA-TBB resin cements to CAD/CAM resin composite. Dent Mater J 2019; 38: 28-32.
- 28) Wattanasirmkit K, Charasseangpaisarn T. Effect of different cleansing agents and adhesive resins on bond strength of contaminated zirconia. J Prosthodont Res 2019; 63: 271-276.
- 29) Cho JH, Kim SJ, Shim JS, Lee KW. Effect of zirconia surface treatment using nitric acid-hydrofluoric acid on the shear bond strengths of resin cements. J Adv Prosthodont 2017; 9: 77-84.
- 30) Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/ cementation to zirconia and other non-silicate ceramics: where are we now? Dent Mater 2011; 27: 71-82.
- 31) Kern M, Wegner SM. Bonding to zirconia ceramic adhesion methods and their durability. Dent Mater 1998; 14: 64-71.
- 32) Okuya N, Minami H, Kurashige H, Murahara S, Suzuki S, Tanaka T. Effects of metal primers on bonding of adhesive resin cement to noble alloys for porcelain fusing. Dent Mater J 2010; 29: 177-187.
- 33) Atsuta M, Matsumura H, Tanaka T. Bonding fixed prosthodontic composite resin and precious metal alloys with the use of a vinyl-thiol primer and an adhesive opaque resin. J Prosthet Dent 1992; 67: 296-300.
- 34) Ali N, Safwat A, Aboushelib M. The effect of fusion sputtering surface treatment on microshear bond strength of zirconia and MDP-containing resin cement. Dent Mater 2019; 35: e107-e112.
- 35) Atsu SS, Kilicarslan MA, Kucukesmen HC, Aka PS. Effect

of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin. J Prosthet Dent 2006; 95: 430-436.

- 36) Luthra R, Kaur P. An insight into current concepts and techniques in resin bonding to high strength ceramics. Aust Dent J 2016; 61: 163-173.
- 37) Okada M, Taketa H, Torii Y, Irie M, Matsumoto T. Optimal sandblasting conditions for conventional-type yttria-stabilized tetragonal zirconia polycrystals. Dent Mater 2019; 35: 169-175.
- 38) Pilo R, Dimitriadi M, Palaghia A, Eliades G. Effect of tribochemical treatments and silane reactivity on resin bonding to zirconia. Dent Mater 2018; 34: 306-316.
- 39) Xie H, Li Q, Zhang F, Lu Y, Tay F, Qian M, et al. Comparison of resin bonding improvements to zirconia between one-bottle universal adhesives and tribochemical silica coating, which is better? Dent Mater 2016; 32: 403-411.
- 40) Iwasaki T, Komine F, Fushiki R, Kubochi K, Shinohara M, Matsumura H. Shear bond strengths of an indirect composite layering material to a tribochemically silica-coated zirconia framework material. Dent Mater J 2016; 35: 461-469.
- Kern M, Thompson VP. Bonding to glass infiltrated alumina ceramic: adhesive methods and their durability. J Prosthet Dent 1995; 73: 240-249.
- 42) Kakura K, Yasuno K, Taniguchi Y, Yamamoto K, Sakai T, Irie A, et al. Zirconia implant with rough surface produced by YAG laser treatment: Evaluation of histomorphology and strength of osseointegration. J Hard Tissue Biol 2014; 23: 77-82.
- 43) Henriques B, Fabris D, Souza JCM, Silva FS, Carvalho Ó, Fredel MC, et al. Bond strength enhancement of zirconiaporcelain interfaces via Nd:YAG laser surface structuring. J Mech Behav Biomed Mater 2018; 81: 161-167.
- 44) Abdullah AO, Muhammed FK, Yu H, Pollington S, Xudong S, Liu Y. The impact of laser scanning on zirconia coating and shear bond strength using veneer ceramic material. Dent Mater J 2019; 38: 452-463.