# Effect of Four Methods of Surface Treatment on Shear Bond Strength of Orthodontic Brackets to Zirconium

Soghra Yassaei<sup>1</sup>, Hossein Agha Aghili<sup>2</sup>, Abdolrahim Davari<sup>3</sup>, Seyed Morteza Saadat Mostafavi<sup>4</sup>

<sup>1</sup>Associate Professor, Department of Orthodontics, Faculty of Dentistry, Shahid Sadoughi University of Medical Sciences, Yazd, Iran
<sup>2</sup>Assistant Professor, Department of Orthodontics, Faculty of Dentistry, Shahid Sadoughi University of Medical Sciences, Yazd, Iran
<sup>3</sup>Associate Professor, Department of Operative Dentistry, Faculty of Dentistry, Shahid Sadoughi University of Medical Sciences, Yazd, Iran
<sup>4</sup>Postgraduate Student, Department of Orthodontics, Faculty of Dentistry, Shahid Sadoughi University of Medical Sciences, Yazd, Iran

#### Abstract

**Objectives:** Providing reliable attachment between bracket base and zirconia surface is a prerequisite for exertion of orthodontic force. The purpose of the present study was to evaluate the effect of four zirconium surface treatment methods on shear bond strength (SBS) of orthodontic brackets.

**Materials and Methods:** One block of zirconium was trimmed into four zirconium surfaces, which served as our four study groups and each had 18 metal brackets bonded to them. Once the glazed layer was removed, the first group was etched with 9.6% hydrofluoric acid (HF), and the remaining three groups were prepared by means of sandblasting and 1W, and 2W Er: YAG laser, respectively. After application of silane, central incisor brackets were bonded to the zirconium surfaces. The SBS values were measured by a Dartec testing machine with a crosshead speed of 1 mm/min. Data were analyzed using one-way ANOVA and Tukey's HSD for multiple comparisons.

**Results:** The highest SBS was achieved in the sandblasted group  $(7.81\pm1.02 \text{ MPa})$  followed in a descending order by 2W laser group  $(6.95\pm0.87 \text{ MPa})$ , 1W laser group  $(6.87\pm0.92 \text{ MPa})$  and HF acid etched group  $(5.84\pm0.78 \text{ MPa})$ . The differences between the study groups were statistically significant except between the laser groups (P<0.05).

**Conclusion:** In terms of higher bond strength and safety, sandblasting and Er: YAG laser irradiation with power output of 1W and 2W can be considered more appropriate alternatives to HF acid etching for zirconium surface treatment prior to bracket bonding.

Keywords: Lasers, Solid-State; Shear Strength; Hydrofluoric Acid; Zirconium

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Corresponding author:M. Mostafavi, Department of

Iran

Orthodontics, Faculty of Den-

tistry, Shahid Sadoughi University of Medical Sciences, Yazd,

Mortezasaadatt@gmailcom

**INTRODUCTION** 

At present, the number of adults seeking orthodontic treatment is increasing. Many of them present to orthodontic clinics with restorations such as crowns and bridges in their mouth, made of yttrium-stabilized tetragonal zirconia (Y-TZP) ceramics or in short, zirconium crowns. These crowns are widely used and favored for their advantages including biocompatibility, aesthetics, cost effectiveness, fracture resistance, and accurate fabrication. Zirconia crowns are used to restore posterior teeth and occasionally anterior teeth when the focus is more on strength rather than aesthetics [1].

Providing reliable attachment between bracket base and zirconia surface is a prerequisite for exertion of orthodontic force. This attachment should be strong enough to prevent bond failure in the course of orthodontic treatment and maintain the uniformity of zirconia following debonding.

Since zirconia is a member of porcelain family, orthodontists use the same methods for preparation of zirconium crowns before bracket bonding as they do for porcelain surfaces [2-4]. Among these methods, the most utilized one is HF acid etching, which has the disadvantages of producing toxic vapors and burning skin and mucous membranes. Furthermore, it may damage the zirconia surfaces. Therefore, finding an alternative method to HF acid etching with fewer side effects on soft tissues and restoration surfaces seems to be necessary. Er: YAG laser irradiation is a new method appreciated by many authors for its advantages in preparing the enamel and porcelain surfaces [2,4,5]. It is a solid laser with a wavelength of 2,940 nm in the infrared range. Sandblasting is another method for preparation of various non- enamel surfaces.

Therefore, the objective of this study was to compare the effect of four methods of zirconium surface preparation, including 10% HF acid etching, Er: YAG laser irradiation with power outputs of 1 and 2 W and sandblasting on SBS of metal brackets to find an appropriate method of zirconia preparation for orthodontic bonding.

## **MATERIALS AND METHODS**

In the present study, one round block of full contour zirconia (yttrium-stabilized tetragonal zirconia ceramic, Zircon Zhan, Prettau, Italy), 95 mm in diameter and 22 mm in height, was used. Using a special burr, the block of full contour zirconia was trimmed into two halfround blocks, which were simultaneously glazed in an oven in a similar fashion. Consequently, we had two half-round blocks, each having two surfaces on their sides.

Thus, we had four zirconia surfaces for bracket bonding. Each of these surfaces served as a study group, prepared differently and had 18 metal brackets bonded to it. Initially the glazed layer of the zirconia was removed using a 0.8 mm round bur. A rectangular outline with a diameter of 12 mm was then marked on zirconia surfaces using nail varnish for bonding of each bracket.

The brackets were arranged so that 10 mm distance was considered between them from each side.

To have the laser operate at its most appropriate power output, four additional square samples of zirconia with a diameter of 16 mm were tested in a pilot study.

The first three specimens were Er: YAG laser (Fontona-1210 Ljubljana, Slovenia) irradiated with an average power output of 1, 2, and 4 W and were compared to the fourth sample, which was prepared using HF acid etching. The four samples were examined under an scanning electron microscope (SEM) (VEGA, Tescan, PA, USA) and considering the burning on the sample lased with 4W Er: YAG (Fig. 1) and the appropriate etching pattern observed in other specimens (Figs. 2-4), it was decided to use the power settings of 1 and 2 W for laser groups in the current study. Regarding surface conditioning, the zirconia surface in group one was etched using 9.6% HF acid (Pulpdent, Watertown, USA). Following twominute application of acid on the zirconium surface, it was washed with a gentle flow of water for 10 seconds and later dried by a blower for 10 seconds.

The zirconia surface in the second group was sandblasted (Renfert, Hilzingen, Germany) with 110  $\mu$ m aluminum oxide particles at 80 Psi pressure for four seconds.

The zirconia surfaces in groups three and four were prepared using Er: YAG laser irradiation. Therefore, group three samples were exposed to 1 W power, 50 MJ energy, 20 Hz frequency and 416 MJ/cm<sup>2</sup> energy density for 60 seconds. The group four samples were exposed to 2 W power, 100 MJ energy, 20 Hz frequency and 832 MJ/cm<sup>2</sup> energy density for 60 seconds.



Fig. 1. SEM micrograph of the burning of the sample lased with 4W Er: YAG laser at ×1000 magnification.



Fig. 2. SEM micrograph of the sample prepared using hydrofluoric acid at ×1000 magnification.

Pattern of movement of laser tip was linear by hand and the time of lasing was calculated with a stopwatch.

The fiber tip of laser headpiece was held at 10 mm distance from the fixed samples.

Then, the surface of the prepared samples was smeared with silane (Pulpdent, Watertown, USA) and dried. Stainless steel standard edgewise maxillary central brackets (Dentsply Gac, NY, USA) were used in this study.



Fig. 3. SEM micrograph of the sample lased with 2 W laser (×1000 magnification)



Fig. 4. SEM micrograph of the sample lased with 1 W laser (×1000 magnification).

The bracket bases were covered with a thin layer of light curing composite resin (Resilience, Ortho Technology Inc., FA, USA) and placed on their pre-specified places.

Once the brackets were placed, curing process was done using 1000 W light emitting diode (LED) light curing unit (Morita, Kyoto, Japan) for 20 seconds (five seconds for each of the occlusal, gingival, mesial, and distal surfaces). The LED tip was held at the closest possible distance to the samples at a 45-degree angle. Before curing of each bracket, the other 17 brackets were covered with aluminum foil to protect them from extra curing. Following bracket bonding, the zirconia samples were stored in water at 37° for 24 hours. They were then subjected to 500 thermal cycles between 5 and 55°C for 30 seconds with a transfer time

of 15 seconds. After thermocycling, debonding was performed using a Dartec testing machine (HC10, Dartec Ltd., Sturbridge, England) with a crosshead speed of 1 mm/min and 0.5 mm blade thickness. For this purpose, samples were fixed and tip of the Dartec testing machine was moved forward at the bracket-zirconium interface until the bond failure. The SBS was then calculated as the maximum force applied divided by the surface area and recorded in megapascals (MPa). The data were confirmed to be normally distributed using the Kolmogorov-Smirnov test. One-way ANOVA and Tukey's post hoc test were used to compare SBS values among groups using SPSS 16.00 software (Microsoft, IL, USA).

## RESULTS

Descriptive statistics including the mean and standard deviation values are presented in Table 1. As shown in Table 1, maximum amount of SBS belonged to group two (sandblast) ( $7.81\pm1.02$  MPa), followed in a decreasing order by group four (2 W laser) ( $6.95\pm0.87$  MPa), group three (1 W laser) ( $6.87\pm0.92$  MPa) and group one (HF acid) ( $5.84\pm0.78$  MPa) (P=0.05).

Furthermore, the narrow range of the confidence interval in the study groups implies that data were not widely scattered. In order to compare the mean values of SBS among the study groups, ANOVA was used, which revealed significant differences among the groups (Table 2).

Multiple comparisons among groups were done by means of Tukey's post hoc test (Table 3), which showed significantly different SBS values among the groups ( $p \le 0.029$ ) except between laser groups (P=0.995).

## DISCUSSION

In this study, the effects of four surface preparation methods on the SBS values of metal brackets to zirconia surfaces were compared. The results of this study revealed that sand-blasted specimens possessed the highest SBS followed by 2W and 1 W Er: YAG laser irradiation and 9.6% HF acid etched groups, respectively.

Surface preparation by laser, known as laser etching, generates heat, which creates porosities on the zirconia surface and provides mechanical retention at the zirconium- composite interface [5].

Table 1.	The mean shear	bond strength of	f brackets to	zirconia	(MPa)	(descriptive	analysis of	the groups)
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Crowna	N	Moon	Std Doviation	Std Ennon	95% Confidence Int	95% Confidence Interval for Mean		
Groups	IN	Wiean	Stu. Deviation	Stu. Error	Lower Bound	Upper Bound		
HF	18	5.8444	.78141	.18418	5.4559	6.2330		
Sandblast	18	7.8122	1.02553	.24172	7.3022	8.3222		
1W Laser	18	6.8789	.92454	.21792	6.4191	7.3387		
2W Laser	18	6.9508	.87670	.20664	6.5149	7.3868		
Total	72	6.8716	1.13142	.13334	6.6057	7.1375		

Fable 2.	ANOVA	of the	samples
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		ANOVA			
SBS	Sum of Squares	Difference	Mean Square	F	Sig.
Between Groups*	35.031	3	11.677	14.215	.000
Within Groups	55.857	68	.821		

\*: The mean difference is significant at the 0.05 level.

In the current study, such etching pattern was observed in electron microscopic examination of lased specimens. Since silane application after different surface conditioning methods increases bond strength [6,7], it was applied to prepared zirconia surfaces before bracket bonding in all four study groups.

As increasing temperature adversely affects the mechanical properties of zirconia ceramics, lower power outputs of Er: YAG laser were used in the current study. Heat generation as a consequence of laser irradiation leads to phase change of ceramics [8]. Higher power outputs cause greater material destruction and are therefore inappropriate for surface conditioning [9]. Our pilot study and electron microscopic examination revealed that 4 W Er: YAG laser irradiation caused burning of zirconia surface and produced micro-cracks on it; while no evidence of micro-crack formation was observed in specimens lased with 1W and 2W laser. A study conducted by Ural and coworkers on the effects of four different sizes of aluminum oxide particles on the bond strength of resin cements to zirconium cores showed that 110 µm particles provided higher bond strength [10]. This finding was further supported by Kulunk and coworkers [11]. Therefore, 110 µm aluminum oxide particles were chosen for sandblasting of the zirconia surface in the current study.

As stated earlier, no previous research has evaluated different zirconium surface preparation methods and their effects on the SBS of brackets. However, similar studies have been done on porcelain crowns. For instance, Yassaei et al. compared the effect of Er: YAG laser etching (with power outputs of 1.6, 2, and 3 W) with 9.6% HF acid etching on SBS of metal brackets to porcelain discs and found insignificant differences between the methods used [4]. However, they did not assess sandblasting. In another study conducted by Akova et al, it was reported that HF acid etching with silane application resulted in the highest bond strength, which again differs from our findings. They also observed that sandblasting and silane application led to higher bond strength compared to Er: YAG laser, which is similar to our result [5].

Ahmad Akhoundi et al. evaluated the tensile bond strength of metal brackets to glazed ceramic surfaces with different surface conditioning techniques including HF acid etching following priming with adhesive and bonding agent alone, and another group was treated with 35% phosphoric acid followed by ceramic primer and adhesive application. They concluded that phosphoric acid can be used instead of HF acid for bonding brackets to the glazed ceramic restorations with adequate tensile bond strength [14].

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.
HF <sup>a</sup>	Sandblast <sup>b</sup>	-1.96778*	.30211	.000
	Laser 1w <sup>c</sup>	-1.03444*	.30211	.006
	Laser 2w <sup>d</sup>	-1.10639*	.30211	.003
Sandblast <sup>a</sup>	$\mathrm{HF}^{\mathrm{b}}$	1.96778*	.30211	.000
	Laser 1w <sup>c</sup>	.93333*	.30211	.015
	Laser 2w <sup>d</sup>	.86139*	.30211	.029
	$\mathrm{HF}^{\mathrm{b}}$	1.03444*	.30211	.006
Laser 1W <sup>a</sup>	Sandblast <sup>c</sup>	93333*	.30211	.015
	Laser 2w <sup>a</sup>	07194	.30211	.995
Laser 2W <sup>a</sup>	$\mathrm{HF}^{\mathrm{b}}$	1.10639*	.30211	.003
	Sandblast <sup>c</sup>	86139*	.30211	.029
	Laser 1w <sup>a</sup>	.07194	.30211	.995

Table 3. Mean difference of shear bond strength among groups (Tukey's test)

\*Groups with the same superscripted letters are not significantly different (P>0.05)

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In our study, we did not use phosphoric acid but we concluded that sandblasting and erbium laser can be used instead of HF acid etching. Another studies by Ahmad Akhoundi et al, also confirms the results of the latter study [12,13].

In another study Ahmad Akhoundi et al. compared conventional orthodontic bonding resin and nano-filled composite. They observed less damage to feldspathic porcelain when the nano-filled composite was used to bond brackets. Thus, they suggested the use of nano-filled composite resins for bonding brackets to feldspathic porcelain restorations [14]. However, it should be noted that the aforementioned studies were conducted on porcelain.

Akin et al. assessed the SBS of zirconia crowns to dental cements and concluded that the specimens lased with 1 W Er: YAG laser had significantly higher bond strength in comparison with the control group [15]. This finding was one of the reasons for choosing Er: YAG laser and 1W power output to prepare the zirconia surfaces in the current study. Murthy et al, also evaluated the SBS of zirconium crowns prepared with five different surface treatments to autopolymerizing resin and concluded that the CO<sub>2</sub> laser caused the highest SBS followed by the HF acid etching and sandblasting with 110 µm alumina [16]. We did not use CO<sub>2</sub> laser and in our study, the SBS of sandblasting was higher than that of HF acid etching.

Kasraei et al. assessed the  $CO_2$  laser surface treatment and concluded that this method significantly increased the SBS of resin cement to zirconia ceramic compared to the control group [17]. Thus, laser irradiation is one of the best methods of surface treatment.

Arami et al. assessed the SBS of the repair composite resin to zirconia ceramic by different surface treatments and concluded that air abrasion with Al<sub>2</sub>O<sub>3</sub> particles was the most effective method for conditioning of zirconia ceramic surfaces and this finding is consistent

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with our result [18]. Although we did not use Nd: YAG laser, Arami et al. showed that Er: YAG laser with 2W power was superior to Nd: YAG laser with 1.5W power in terms of efficacy [18].

Uludamar et al. showed that sandblasted zirconia crowns had higher SBS compared to Er: YAG irradiated samples and also bur conditioned specimens. This finding is corroborated by the results of our study; although we did not have bur-conditioned samples [19].

Akyil et al. evaluated nine different surface treatment methods on 141 zirconia samples and found that the highest bond strength between resin cements and zirconia crowns was achieved with sandblasting and silane application. They stated that Er: YAG and CO<sub>2</sub> lasers can be suitable alternatives to sandblasting [20] and this is further supported by the results of the current study. In a similar study, Cavalcanti concluded that higher SBS to resin cement could be attained using sandblasting plus metal primer application compared to Er: YAG laser irradiation [8]. As mentioned earlier, all the above-mentioned studies evaluated the bond strength of zirconia crowns to dental cements or enamel surfaces and no research was found on the SBS of orthodontic brackets to zirconium restorations.

## Limitations

Because of economic issues and limitations due to sanction, zirconium blocks were used instead of 72 separate zirconium samples. The present study showed that using one block of zirconium and trimming it into four surfaces for bracket bonding not only had no adverse effects on the results, but also produced a uniform surface.

## CONCLUSION

The results of the present study showed that sandblasting of zirconium surface led to the highest SBS values in bracket bonding followed by 2W laser, 1W laser, and HF acid etching in a descending order. It was concluded that zirconium surface treatment with sandblasting and 1W and 2W laser irradiation was safe and provided higher SBS values in comparison with HF acid etching. Lower power outputs of laser did not cause zirconium surface damage and provided appropriate SBS values.

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