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Effects of Different Surface Treatment Methods and Zirconia Primers on Shear Bond Strength of Orthodontic Brackets to Zirconium

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Objectives Providing a reliable attachment between the bracket base and zirconia surface is a prerequisite for exertion of orthodontic forces. The purpose of the present study was to evaluate the effect of two surface treatment methods and three primers on shear bond strength (SBS) of orthodontic brackets to zirconia surface.
Methods Zirconia blocks were milled and embedded in acrylic resin. The polished zirconia surfaces were randomly prepared with sandblasting (SB) and Er:YAG laser application (LA). Each group of 45 (SB and LA) was further divided into 3 subgroups of 15. The subgroups received different primers namely Z-Prime Plus, MKZ primer and Clearfil SE Bond Primer. The SBS values were measured and compared using two-way ANOVA. SPSS 21 for Macintosh was used for all statistical analyses. Level of significance was set at $P<0.05$.
Results The SB group exhibited a mean SBS of 14.393 MPa, which was significantly higher than the mean SBS recorded for LA group (5.683 MPa; $P<0.05$). In SB subgroups, there was no significant difference among the primers in SBS ($P= 0.391$), but this was not the case for laser subgroups ($P< 0.05$) and the subgroups that received Clearfil SE and Z-Prime Plus had higher SBS than the MKZ primer subgroup.
Conclusion This study suggests that simultaneous use of sandblasting and primers containing 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer can result in acceptable SBS of brackets to zirconia surfaces.
Keywords Zirconium; Lasers, Solid-State; MDP adhesion promoting monomer; Shear Strength

Introduction

In the recent years, there has been a steady increase in the number of adult patients who seek orthodontic treatment. Regarding the fact that adults usually have restored teeth in their oral cavity, the need to bond brackets to a variety of materials has emerged.[1] Among these materials, ceramics which are widely used in dentistry present particular bonding problems due to their inert nature.[2-4] Therefore, different approaches have been proposed to provide acceptable bond strength between ceramics and orthodontic brackets. Such preparation methods can be classified into mechanical, chemical, and combined methods.[4]

Mechanical approaches roughen the ceramic surface by removing the glaze. Surface roughening can be performed through various methods such as milling, sandblasting, tribochemical preparation, and use of lasers.[5] Although some studies showed a significant increase in bond strength following these methods, other studies reported unsatisfactory bond strength.[6, 7, 8] Besides its controversial effectiveness, roughening of ceramic surfaces results in higher incidence of crack initiation and porcelain fracture associated with debonding.[2, 9-12]

Application of high-power lasers is one of the recent techniques to create surface porosities in ceramics and prepare them for bonding. Laser-treated surfaces of ceramics have shown favorable micromechanical retention patterns, making this method an attractive alternative to other surface treatment methods.[13, 14]

Preparation of ceramic surfaces for bonding with chemical methods can be achieved by the use of hydrofluoric acid.[15] Although the bond strength obtained with hydrofluoric acid etching is satisfactory, it is a harmful and irritating compound for the soft tissue.[16]

In the recent years, zirconium has gained major popularity among all-ceramic systems due to its mechanical and optical properties.[17] However, unlike silica ceramics, zirconia ceramic cannot be etched with hydrofluoric acid, since it lacks silica in its structure.[18] Also, mechanical surface preparation alone is not sufficient to achieve acceptable bond strength to zirconia surfaces and should be combined with chemical surface treatment methods. Ten-methacryloyloxydecyl dihydrogen phosphate (MDP)-containing primers are suggested for this purpose. MDP acidic groups (phosphoric acid) form chemical bonds with the oxide layer of zirconia.[15, 19]

Although various surface treatment methods have been proposed to overcome bonding problems with zirconium, a consensus has not been reached yet, and data regarding the problem of bonding of orthodontic brackets to zirconia surface are still lacking. Therefore, the purpose of this study was to evaluate the effect of two surface treatment methods and three primers on shear bond strength (SBS) of orthodontic brackets to zirconium surface.

Methods and Materials

In this in vitro experimental study, 90 zirconia blocks (3M™ Lava™ Premium, Saint Paul, Minnesota, USA) were cut into blocks measuring 2 x 6 x 6 mm (post-sinter dimensions) using a computer-aided design/computer-aided manufacturing system, and then polished with 600-grit silicon carbide paper. After grinding, the specimens were sintered according to the manufacturer's protocol. Each specimen was then ground with abrasive paper with 35 µm diamond particles to produce a uniformly smooth and flat bonding surface. Additionally, the specimens had chamfered borders to facilitate retention in acrylic mounting. The blocks were then embedded in acrylic resin (Buehler, Lake Bluff, IL, USA). The polished zirconia surfaces were randomly prepared with sandblasting (SB) and Er:YAG laser (LA). Forty-five samples were prepared by sandblasting. For this purpose, the intraoral micro-blasting unit (Danville Microetcher, Carlsbad, CA, USA) was set at 40 Psi, and the specimens were abraded for 15 seconds with 50-µm aluminum oxide particles (Danvile Materials, Carlsbad, CA, USA) at 5-10 mm distance in an area approximately 2-3 times larger than the orthodontic bracket base. The remaining 45 specimens were prepared with Er:YAG laser (Deka, Calenzano, Italy) irradiation. Accordingly, the specimens were lased with 2 W power, 100 MJ energy, 20 Hz frequency and 832 mJ/cm^2 energy density for 60 seconds.[20]

Each group of 45 (SB and LA) was then further divided into 3 subgroups of 15. The 3 subgroups received different primers namely Z-Prime Plus (Bisco, Schaumburg, IL, USA), MKZ primer (Bredent, Serden, Bavaria, Germany) and Clearfil SE Bond Primer (Kuraray, New York, NY, USA) (SE).

The primers were used according to the manufacturers' recommendations. A standard lower incisor orthodontic bracket (American Orthodontics, Sheboygan, WI, USA) was bonded by one single operator with 150 g even pressure using an ergometer (Dentaurum, Ispringen, Germany) to determine the amount of pressure. Transbond XT was used as adhesive. Excessive adhesive was removed. The adhesive was cured from the mesial, distal, incisal, and gingival sides for 5 seconds each, using a LED curing unit (Morita, Kyoto, Japan). The specimens were thermocycled in a thermocycler (Dorsa, Tehran, Iran) for 1000 cycles at 5-55°C with 20 seconds of dwell time in each water bath and 30 seconds of transfer time. Then, the specimens were incubated at 37°C for 24 hours.

SBS testing was carried out using a universal testing machine (Instron Z020; Zwick Roell, Ulm, Germany). Load was applied at a crosshead speed of 0.5 mm/minute perpendicular to the interface of the bonded area, until debonding occurred. The universal testing machine showed the debonding load in Newtons (N); the values were divided by the surface area to report the SBS in megapascals (MPa).

Following the process of debonding, the surface of zirconia and the base of brackets were inspected under a stereomicroscope (Olympus SZX9, Tokyo, Japan) at x10 magnification. To assess the location of debonding, the Bordeaux classification was used as follows:[21]

Type I: Failure at the adhesive-bracket base interface: 90% or greater of the bracket base is exposed, and 10% or less of the zirconia surface is free from adhesive.

Type 2: Combined failure at the adhesive-bracket base interface and the zirconia-adhesive interface: Less than 90% but more than 10% of the bracket base is exposed, or more than 10% but less than 90% of the zirconia surface is free from adhesive.

Type 3: Failure at the zirconia-adhesive interface: 10% or less of the bracket base is exposed, and 90% or more of the surface of zirconia is free from adhesive.

Type 4: Fracture of the bracket itself. Fracture of the bracket during removal leaving a portion of the bracket still bonded to zirconia.

Type 5: Fracture of the zirconia ceramic itself. A portion of the zirconia is removed with the bracket base without loss of more than 10% of the adhesive from the bracket base.

Statistical analysis:

Normal distribution of the SBS data was confirmed by the Kolmogorov-Smirnov test, and the equality of variances was assessed by the Levene's test. The SBS values were compared using two-way ANOVA. Level of significance was set at $P<0.05$. SPSS version 21 for Macintosh was used for all statistical comparisons.

Results

Descriptive statistics including the mean SBS of different groups and standard deviation values are presented in Table 1.

Table 1- Mean SBS of different groups and standard deviation values

Surface Treatment	Primer	Mean	Std. Deviation	Minimum	Maximum
SB	ZP	14.2807	2.58988	10.78	18.39
	MKZ	15.1185	2.52692	9.44	18.56
	SE	13.7963	2.80712	10.11	17.89
LA	ZP	6.7370	2.12645	3.89	10.56
	MKZ	2.6519	.86563	1.61	4.44
	SE	7.6778	2.42190	3.89	11.33

SB: Sandblasting, ZP: Z-Prime Plus, MKZ: MKZ primer, SE: Clearfil SE Bond primer, LA: Laser application

The mean SBS in SB and LA groups was compared using two-way ANOVA. The SBS was significantly higher in the SB group ($P<0.05$). For further investigation, one-way ANOVA was applied to compare the subgroups of each surface treatment method with each other. In the SB group, different primers did not show statistically significant differences in SBS ($P= 0.391$). However, in the LA group, the primer affected the SBS values ($P<0.05$). Pairwise comparisons between different primers in the LA group showed that Clearfil SE and Z-Prime plus had higher SBS than MKZ primer ($P<0.05$).

The most common type of failure related to each group is shown in Figure 1.

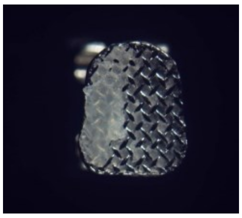
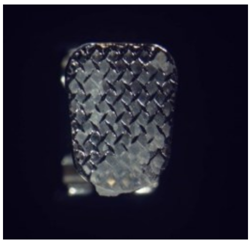
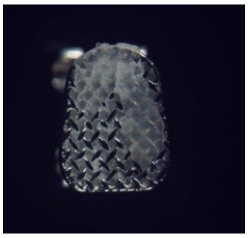
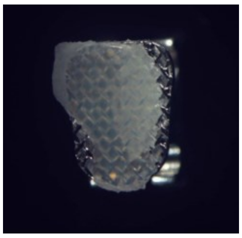
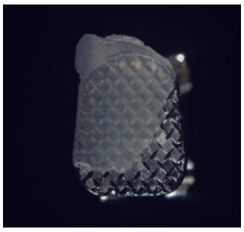
Group	Most common type of failure	Microscopic image
SB+ ZP	Type II	
SB+MKZ	Type I	
SB+SE	Type II	
LA+ZP	Type III	
LA+MKZ	Type III	
LA+SE	Type III	

Figure 1: Most common type of failure related to each group
SB: Sandblasting, ZP: Z-Prime Plus, MKZ: MKZ primer, SE: Clearfil SE Bond primer, LA: Laser application

Discussion

The use of zirconia ceramics is steadily increasing in the recent years. The mechanical and optical properties of zirconia satisfy dentists and patients, but achieving a reliable bond to zirconia surfaces comes with difficulty.[22, 23]
In this study, the effects of two surface preparation methods and three zirconia primers on SBS of metal brackets to zirconia surfaces were compared. According to the results, there was a significant difference between the SB and LA groups regarding SBS values. All 3 subgroups in SB group exhibited significantly higher SBS than the LA group.
A previous study reported that SBS in the range of 5.9 to 7.8 MPa was sufficient to withstand normal oral masticatory and orthodontic forces.[24] In this study, the only group which showed a SBS below this acceptable range was the subgroup which was treated with laser and received MKZ primer.
The results of stereomicroscopic evaluation were in line with the data from SBS test, as they showed higher frequency of types I and II fracture in the SB group and type III fracture in the LA group, and confirmed weaker bond in the LA group.
In this study, sandblasting was performed for 10 seconds with 50 µm alumina particles from a 10 mm distance. Most studies used the same size of particles for this purpose, but for different times and distances.[25, 26]
Different lasers may be used for surface treatment by surface roughening, and there is controversy about the effectiveness of laser irradiation for enhancement of SBS of zirconia ceramic.[13, 27]
In a recent study, Hatami et al. evaluated the effects of three lasers (Nd:YAG, CO2 and Er:YAG) on SBS of zirconia and showed that Er:YAG laser irradiation was the most effective treatment on bond strength comparable to sandblasting.[28] However several other studies investigating the bond strength and durability of various bonding methods to high-strength ceramics indicated that sandblasting led to higher bond strength than laser treatment.[18, 29, 30] This study was designed to address the controversy in this area, and our results indicated that sandblasting led to higher bond strength than laser treatment.
An additional approach to achieve higher bond strength to zirconia is to chemically condition its surface. The proposed chemical treatment methods include silanization, acid etching, and the use of primers containing phosphate and carboxylic acid.[31] The sole application of the first two methods has failed to achieve acceptable bond strength to zirconia.[32] Therefore, great attention has been directed towards conditioning of zirconia with chemical primers. Studies indicate that carboxyl-based primers such as 4-methacryloyloxyethyl trimellitate anhydride are unable to chemically bond to zirconia. However, 10-MDP has shown promising results. MDP has a phosphoric-acid group at one end with an affinity for metal oxides such as zirconia, and a

vinyl group at the other end which bonds to the resin matrix. The combination of sandblasting and use of MDP containing primer has been suggested in order to achieve a hydrothermally durable bond to zirconia.[33, 34]

In a study conducted by Byeon et al, the effects of sandblasting and MDP containing primers was assessed on the bond between zirconia and orthodontic metal brackets. The highest SBS (13.36 MPa) was achieved in the group which received sandblasting and MDP containing silane primer and was close to the mean SBS of SB group in this study (14.393 MPa). The lowest SBS was seen in the group which was only polished, followed by the sandblasted group. The results emphasized on the importance of both mechanical and chemical treatments of zirconia surface for an acceptable bond to brackets.[35]

Yassaei et al. performed a study to compare the bond strength between brackets and ceramic surfaces after preparation by laser, sandblasting, and hydrofluoric acid application. Their study revealed that the highest bond strength was achieved following preparation by sandblasting, which is in line with our results. Although laser treated groups showed significantly lower bond strength than the sandblasted group, the SBS was reported to be enough to withstand orthodontic forces. The highest bond strength in their study was 7.8 MPa which was lower than the value in the present study (15.11 MPa); the lower SBS may be due to the use of Pulpodent as primer, which lacks MDP monomer. These findings point out the importance of MDP monomer in achieving higher bond strength to ceramic surfaces.[36]

It should be noted that this was an in vitro study. In vivo studies and further investigations may be necessary to confirm that our bonding protocols are repeatable under clinical conditions. Factors such as saliva, water, or oil contamination, oral temperature changes, and masticatory forces other than shear forces along with other factors could alter the bonding of orthodontic brackets to zirconia in the long-term. Other areas of research and interest to clinicians may be the long term effects of micromechanical abrasion to zirconia restorations, adhesive removal/polishing effects on zirconia, and whether or not there are any esthetic drawbacks to these procedures after bracket removal.

Conclusion

This study suggests that ideal bracket bonding to zirconia surfaces would include a method of sandblasting followed by the application of a primer which contains MDP monomer.

Conflict of Interest

No Conflict of Interest Declared ■

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