Original Article / Özgün Araştırma

Effect of Different Surface Treatments on the Surface Roughness and Orthodontic Bond Strength of Partially-stabilized Zirconia

Farklı Yüzey İşlemlerinin Parsiyel Stabilize Zirkonyanın Yüzey Pürüzlülüğü ve Ortodontik Bağlanma Dayanımına Etkisi

Mustafa Borga Dönmez[1], Betül Ballı Demirel[2], Münir Demirel[3], Yasemin Gündoğdu[4], Hamdi Şükür Kılıç[5]

[1]İstinye University Faculty of Dentistry, Department of Prosthodontics, İstanbul, Turkey
[2]İstinye University Faculty of Dentistry, Department of Orthodontics, İstanbul, Turkey
[3]Biruni University Vocational School, Department of Oral and Dental Health, İstanbul, Turkey
[4]Selçuk University Kadınhanı Faik İçil Vocational High School, Department of Computer Technologies, Konya, Turkey
[5]Selçuk University Faculty of Science, Department of Physics, Konya, Turkey

Abstract

Objective: To investigate the effect of different surface treatments on the surface roughness (R_a) and shear bond strength (SBS) of partially stabilized zirconia (Y-PSZ) with different yttrium content.

Materials and Methods: Zirconia samples were milled from 5Y-PSZ and 4Y-PSZ disks and divided into 5 groups: Control (C), sandblasting (APA), single-step self-etch primer etching (MEP), Er:YAG laser (ER), and femtosecond laser (FS) irradiation (n=11). Surface R_a was measured and metallic mandibular incisor brackets were bonded. SBS test was performed after thermocycling. Data were analyzed using two-way ANOVA and Tamhane's T2 tests (α=0.05).

Results: Only surface treatment affected R_a and SBS (p<0.001). FS groups had the highest (p<0.001), and C groups had the lowest values R_a (p≤0.001). MEP groups had lower R_a than ER and APA (p≤0.002). FS and APA groups resulted in the highest SBS (p<0.001), while MEP groups achieved significantly higher SBS than ER groups (p<0.001). The C group showed the lowest SBS (p<0.001).

Conclusion: Within the limitations of this *in vitro* study, sandblasting and FS irradiation was found to be the most effective surface treatments for metallic bracket bonding to Y-PSZ.

Keywords
ARI, femtosecond laser, Monobond Etch & Prime, partially stabilized zirconia

Anahtar Kelimeler
ARI, femtosaniye lazer, Monobond Etch & Prime, parsiyel stabilize zirkonya

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Address for Correspondence/Yazışma Adresi: Assist. Prof. Mustafa Borga Dönmez, DDS, PhD, İstinye University Faculty of Dentistry, Department of Prosthodontics, İstanbul, Turkey
Phone : +90 850 283 60 21
E-mail : borga.donmez@istinye.edu.tr
ORCID ID: orcid.org/0000-0002-3094-7487

Öz

Amaç: Bu çalışmanın amacı, farklı yüzey işlemlerinin parsiyel stabilize zirkonyanın (Y-PSZ) yüzey pürüzlülüğü (R_a) ve makaslama bağlanma dayanımına (SBS) olan etkisini değerlendirmektir.

Gereç ve Yöntemler: Zirkonya örnekler, 5Y-PSZ ve 4Y-PSZ disklerinden frezeleme ile hazırlandı ve beş gruba ayrıldı: Kontrol (C), kumlama (APA), tek aşamalı kendiliğinden pürüzlendiricili primer ile pürüzlendirme (MEP), Er:YAG lazer (ER) ve femtosaniye lazer (FS) ışınlama (n=11). R_a ölçüldü ve metalik mandibular kesici braketler yapıştırıldı. SBS testi, ısıl döngüden sonra gerçekleştirildi. Veriler, iki-yönlü ANOVA ve Tamhane T2 testleri ile analiz edildi (α=0,05).

Bulgular: R_a ve SBS sadece yüzey işlemlerinden etkilenmiştir (p<0,001). FS grupları en yüksek R_a'yı (p<0,001), C grupları ise en düşük R_a'yı göstermiştir (p≤0,001). MEP grupları, ER ve APA'dan daha düşük Ra sonuçları vermiştir (p≤0,002). FS ve APA

grupları en yüksek SBS ile sonuçlanırken (p<0,001), MEP grupları ER gruplarından önemli ölçüde daha yüksek SBS elde etmiştir (p<0,001). C grupları ise en düşük SBS'yi göstermiştir (p<0,001).
Sonuç: Bu *in vitro* çalışmanın sınırlamaları dahilinde, Y-PSZ metalik braket bağlanması için APA ve FS ışınlamasının en etkili yüzey işlemleri olduğu bulunmuştur.

Introduction

Monoclinic, tetragonal, and cubic are 3 different crystallographic structures of zirconia (1). Dental zirconia usually contains 3 mol% of yttrium oxide, which stabilizes the tetragonal phase (2,3). Even though zirconia has exceptional physical properties, it is opaque (3,4) and technical complications have been reported (1,5). Monolithic zirconia, which has reduced alumina content was introduced to overcome these problems (6,7). Monolithic zirconia has better translucency (3), yet it still lacks the translucency that glass-ceramics present (6). Recently introduced partially stabilized zirconia (Y-PSZ) (3) has increased yttrium oxide that introduced the cubic phase along with the tetragonal phase (2,6,8). Cubic phase reduces the light scattering at the borders of zirconium dioxide crystals resulting in a more translucent material (6).

Orthodontic treatment and esthetic dental restorations are increasing their popularity among adult patients (9-12). However, traditional adhesives are unsatisfactory to maintain adequate bonding between the porcelain surface and the orthodontic bracket (13). Various lasers have been suggested for bracket bonding (14,15). Among them, Ti:sapphire femtosecond laser (FSL), which emits ultrashort pulses (1 fs $=10^{-15}$ s) not only produces a clean surface, but also reduces phase transformations (15) and have a slight heating effect (9). Several studies investigated the effect of FSL on the shear bond strength (SBS) of orthodontic brackets (9,16,17). However, to authors' knowledge, no study has examined the impact of lasers on the SBS of metal brackets to Y-PSZ. Moreover, a single-step self-etching ceramic primer [Monobond Etch & Prime (MEP); Ivoclar Vivadent, Schaan, Liechtenstein] has been launched recently and the number of the studies on the effect of this material on zirconia are limited (11,13,18). Therefore, the purpose of this study was to investigate the effect of different surface treatments on the surface roughness (R_a) and SBS of Y-PSZ with different yttrium content. The null hypotheses were that surface treatments and material type would not affect R_a or SBS.

Materials and Methods

One hundred and ten specimens were milled (CEREC inLab MC X5; Dentsply Sirona, Bensheim, Germany) from 5 mol% and 4 mol% (Ceramill Zolid FX and Zolid HT+; Amann Girrbach, Pforzheim, Germany) zirconia discs and sintered in a furnace (1450 °C, 8 h, Ceramill Therm; Ivoclar Vivadent, Schaan, Liechtenstein). Specimens were then polished with silicone-carbide papers (#600, 800, and 1000) to final dimensions (12x12x1.5 mm), embedded in auto-polymerizing acrylic resin (SC; Imicryl, Konya, Turkey), and divided into 5 groups (n=11):

Group C: No treatment

Group APA: Sandblasting with 50 µm Al_2O_3 (Korox; BEGO, Bremen, Germany) particles at 2 bar pressures from 10 mm distance for 10 s (2).

Group MEP: MEP etching for 60 s. A micro-brush was used to apply the product for the first 20 s. After 60 s, specimens were rinsed and air-dried.

Group Er:YAG laser (ER): ER laser beams (Fotona; AT Fidelis, Ljubljana, Slovenia) delivered perpendicularly approximately from 2 mm were used to irradiate specimens with a non-contact hand piece (R02) that has an integrated spray nozzle (Wavelength: 2940 nm, frequency: 10 Hz, pulse duration: 10 s, pulse width: 100 µs, energy level: 400 mJ, power: 4 W).

Group FS: FSL consists of two basic units, one of which is oscillator seed laser (Quantronix, Ti-Light, NY, USA) that produces 3 nJ per laser pulse energy with 85 MHz repetition rate at the wavelength of 800 nm. Another unit amplifier laser operates at 1-3 kHz repetition rate with 3.5 mJ per pulse with the fundamental laser wavelength at 800 nm (Quantronix, Integra-C-3.5, NY, USA). The micromachining unit (Quantronix, Q-Mark, NY, USA) capable of operating in accordance with the FSL and the system that can be controlled by computer. F-theta lens was focused on the specimen from 11 cm. In the present study, a marking speed with 10 mm/s, skip speed with 125 mm/s and the repetition with five times were carried out in 10 mm x 10 mm square for each zirconia specimen. All specimens were ablated using 200 mW laser power and 9.819 10^{13} W/cm^2 laser intensity to create a checkered pattern.

Specimens were cleaned ultrasonically for 10 min (Whaledent Biosonic; Whaledent Inc., New York, USA). R_a of the specimens were measured from 5 different regions with a 2-dimensional contact profilometer (MarSurf PS1; Mahr GmbH, Göttingen, Germany) and these values (µm) were averaged. One additional specimen from each group was prepared as described and observed with scanning electron microscope (SEM) (EVO LS-10; Zeiss, Cambridge, UK) at 25 kV (500× and 1000× magnifications).

A light-polymerized adhesive primer (Tranbond XT Primer; 3M Unitek, Monrovia, California, USA) was applied to pretreated zirconia surfaces and mandibular incisor orthodontic metal brackets (Mini Master; American Orthodontics, Sheboygan, Wisconsin, USA), which had an average surface area of 10.37 mm^2 were bonded by using an adhesive resin (Transbond XT; 3M Unitek, Monrovia, California, USA). Excess cement was removed with an explorer and the specimens were irradiated with an LED curing unit (Bluephase; Ivoclar Vivadent, Schaan, Liechtenstein) from the occlusal and the gingival bracket edges for 20 s at 1200 mW/cm^2 intensity. Bonding was performed by a single experienced clinician (B.B.D.). Specimens were then subjected to thermocycling (5,000 cycles at 5-55 °C, dwell time of 15 s) to replicate an intraoral period of relatively six months (19).

A universal testing machine (Mod Dental Micro Shear Tester; Esetron Smart Robotechnologies, Ankara, Turkey) with a crosshead speed of 0.5 mm/min was used for SBS tests. The knife-edge rod was positioned perpendicular to the edge of the brackets' base. SBS (MPa) was calculated by using the equation:

SBS (MPa) = Maximum load (N) / Surface area (mm^2)

Adhesive remnant index (ARI) was determined by using a stereomicroscope (Olympus SZ61; Olympus Corp, Tokyo, Japan) at 45× magnification to assess the failure modes. One sample from each group exhibiting the dominant ARI score was analyzed by using SEM (64×). The scores for ARI were as follows (20):

Score 0: No adhesive on the specimen,

Score 1: Less than 50% of adhesive on the specimen,

Score 2: More than 50% of adhesive on the specimen,

Score 3: All adhesive on the specimen, with distinct impression of the bracket mesh.

Statistical Analysis

Data were analyzed (SPSS 23, SPSS Inc; Chicago, IL, USA) by using 2-way analysis of variance (ANOVA) and Tamhane's T2 tests. In addition, the correlation between these parameters was evaluated with Pearson's correlation analysis (α=0.05). Number of specimens was decided based on a power analysis (power: 0.80, α: 0.05, and effect size: 0.4).

Results

Surface treatments had a significant effect on R_a and SBS ($p<0.001$), whereas the effect of material type ($p\geq0.192$) and the interaction between the main factors ($p\geq0.312$) were nonsignificant for both parameters. For 4Y-PSZ, FS group (4.87±0.41) had the highest R_a ($p<0.001$), whereas C group (0.24±0.04) showed the lowest ($p\leq0.001$). APA (0.49±0.05) and ER (0.47±0.06) groups had similar values ($p=0.997$) that were higher than that of MEP group (0.36±0.04) ($p\leq0.002$). For 5Y-PSZ, FS group (5.13±0.62) had the highest ($p<0.001$) and C group (0.26±0.07) had the lowest R_a ($p\leq0.001$). APA (0.48±0.04) and ER (0.47±0.05) groups had similar values ($p>0.05$) that were higher than that of MEP (0.39±0.04) group ($p\leq0.006$).

For 4Y-PSZ, FS (19.88±2.69) and APA (18.74±2.38) groups had the highest ($p<0.001$) and C group (3.46±0.47) had the lowest SBS ($p<0.001$). MEP group (13.27±2.39) had higher SBS than ER group (5.42±0.59) ($p<0.001$). For 5Y-PSZ, FS (20.58±2.47) and APA (19.47±2.63) groups had the highest SBS values ($p<0.001$). MEP group (12.83±2.46) had higher SBS than ER group (5.15±0.82) ($p<0.001$). C group (2.82±0.38) had the lowest values ($p<0.001$). Pearson correlation analysis showed that there was no significant correlation between R_a and SBS for any of the groups ($p\geq0.154$).

Figures 1 and 2 depict surface alterations after surface treatments. In general, surface treatments resulted in similar surface modifications for both materials. C groups showed little to no irregularities, while MEP etching led to somewhat similar surfaces with more pronounced grooves caused by the etchant. APA generated an irregular topography and roughness of the treated surfaces was apparent. ER treatment was characterized with concave and convex areas, and evident microcrack formation. FS groups presented the most precise surface change with clear, square shaped depressions and no microcracks.

Representative SEM images of the bond failures are presented in Figures 3 and 4, while bond failure types (n and %) are shown in Table 1. In both C groups, 100% of the specimens demonstrated score 0. While the failure types of ER and MEP treated specimens were scores 0 and 1, APA and FS groups showed scores 2 and 3.

Discussion

Even though material type was not effective, surface treatments resulted in significant differences

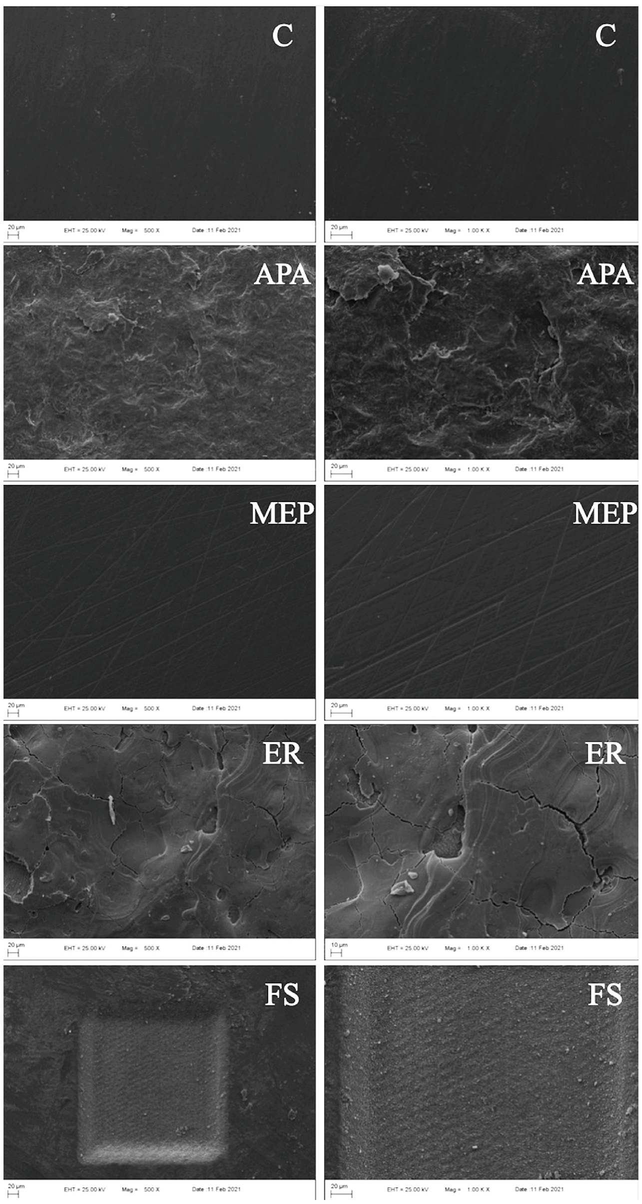

Figure 1. SEM images (500× and 1000×) of 4Y-PSZ specimens after surface treatments

C: Control, APA: Sandblasting, MEP: Monobond Etch & Prime etching, ER: Er:YAG laser irradiation, FS: Femtosecond laser irradiation, SEM: Scanning electron microscope, Y-PSZ: Partially stabilized zirconia

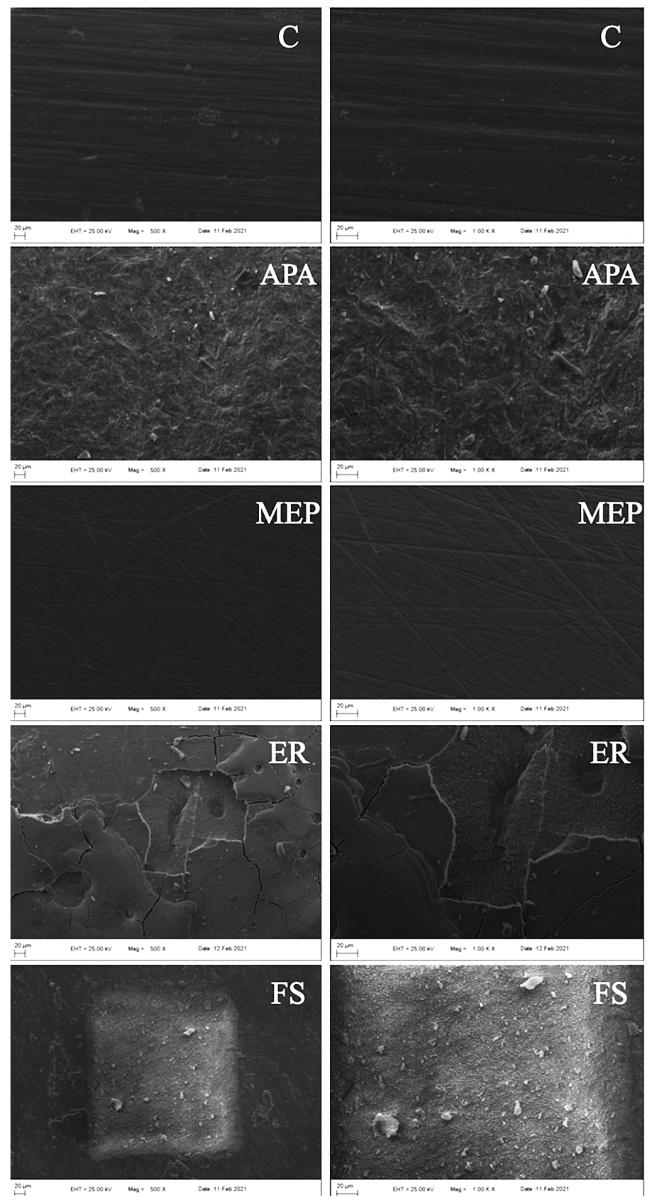

Figure 2. SEM images (500× and 1000×) of 5Y-PSZ specimens after surface treatments

C: Control, APA: Sandblasting, MEP: Monobond Etch & Prime etching, ER: Er:YAG laser irradiation, FS: Femtosecond laser irradiation, SEM: Scanning electron microscope, Y-PSZ: Partially stabilized zirconia in SBS and R_a. Therefore, the null hypotheses were rejected.

Previous studies have shown that zirconia had higher R_a after FSL irradiation when compared with APA (1,9). The result of this study supports this finding as the specimens of FS groups had the highest R_a. SEM images of FS groups revealed the conspicuous surface alteration with precise square shaped depressions, which were also evident to naked eye. These depressions contributed to a greater surface area that orthodontic resin cement penetrated, which may have led to greater SBS values. R_a results of MEP treated specimens were inferior to those treated with APA, ER, and FS. As seen in SEM images, MEP treated

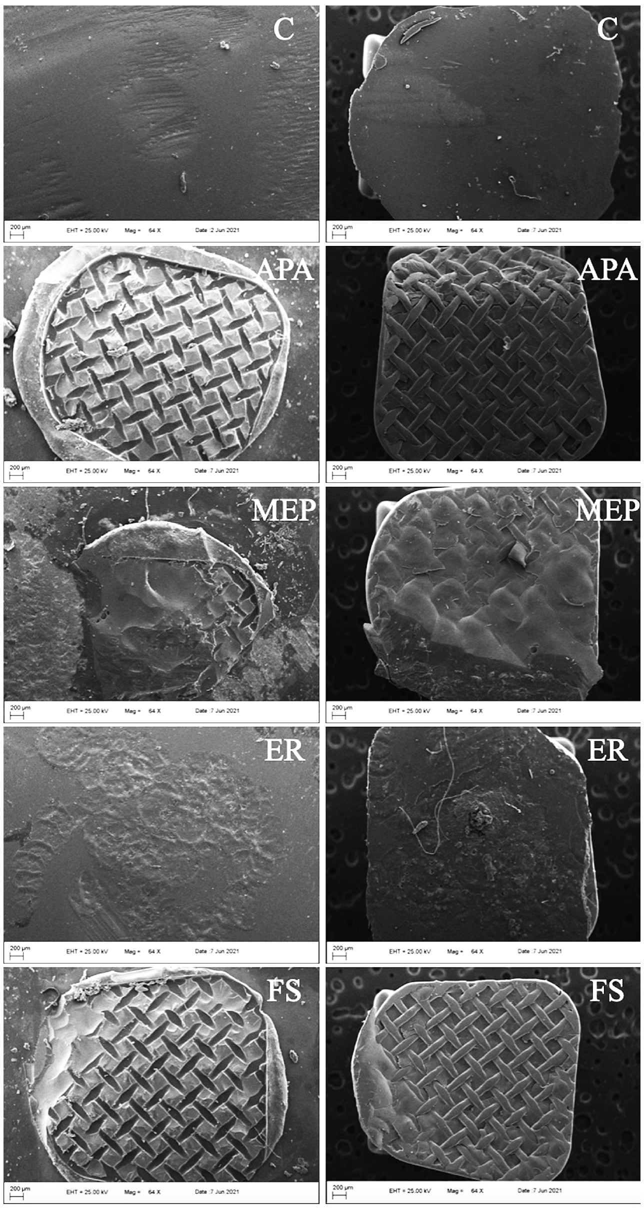

Figure 3. SEM images (64×) of the debonded 4Y-PSZ specimens and metallic bracket surfaces after SBS test
C: Control, APA: Sandblasting, MEP: Monobond Etch & Prime etching, ER: Er:YAG laser irradiation, FS: Femtosecond laser irradiation, SEM: Scanning electron microscope, Y-PSZ: Partially stabilized zirconia, SBS: Shear bond strength

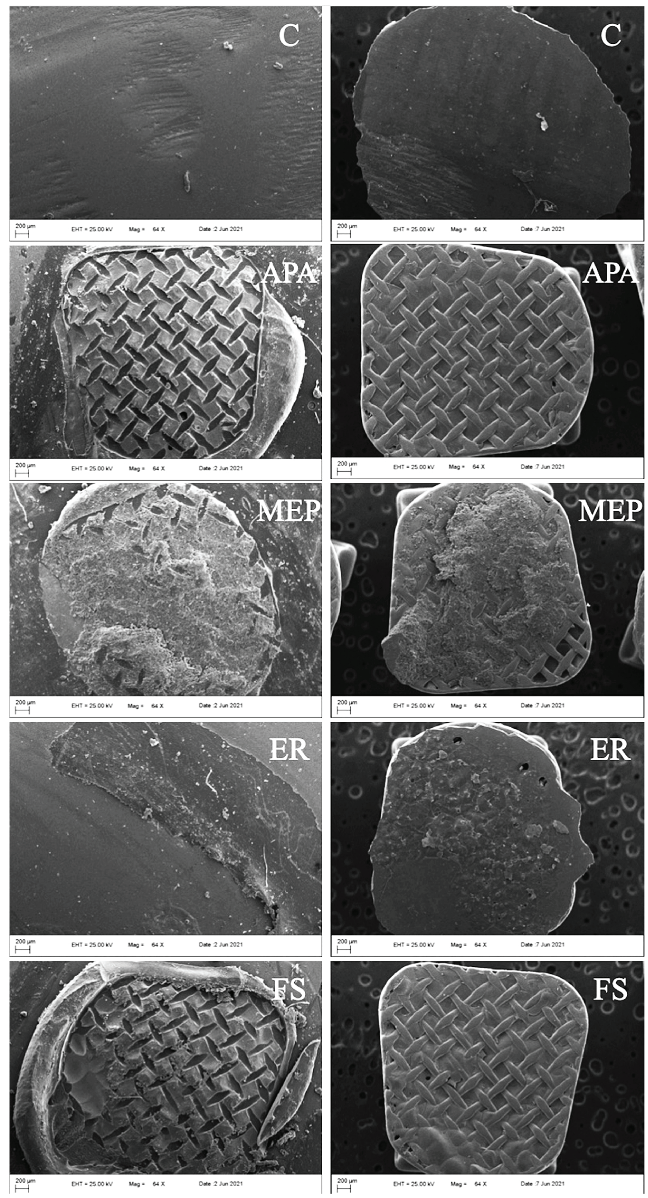

Figure 4. SEM images (64×) of the debonded 5Y-PSZ specimens and metallic bracket surfaces after SBS test
C: Control, APA: Sandblasting, MEP: Monobond Etch & Prime etching, ER: Er:YAG laser irradiation, FS: Femtosecond laser irradiation, SEM: Scanning electron microscope, Y-PSZ: Partially stabilized zirconia, SBS: Shear bond strength

Table 1. Bond failure mode scores (ARI) (n and %)

	C		APA		MEP		ER		FS	
	4 Y-PSZ	5 Y-PSZ	4 Y-PSZ	5 Y-PSZ	4 Y-PSZ	5 Y-PSZ	4 Y-PSZ	5 Y-PSZ	4 Y-PSZ	5 Y-PSZ
Score 0	11 (100%)	11 (100%)	0 (0%)	0 (0%)	4 (36%)	3 (27%)	5 (45%)	3 (27%)	0 (0%)	0 (0%)
Score 1	0 (0%)	0 (0%)	0 (0%)	0 (0%)	6 (55%)	6 (55%)	6 (55%)	8 (73%)	0 (0%)	0 (0%)
Score 2	0 (0%)	0 (0%)	3 (27%)	3 (27%)	1 (9%)	2 (18%)	0 (0%)	0 (0%)	2 (18%)	3 (27%)
Score 3	0 (0%)	0 (0%)	8 (73%)	8 (73%)	0 (0%)	0 (0%)	0 (0%)	0 (0%)	9 (82%)	8 (73%)

ARI: Adhesive remnant index, C: Control, APA: Sandblasting, MEP: Monobond Etch & Prime etching, ER: Er:YAG laser irradiation, FS: Femtosecond laser irradiation, Y-PSZ: Partially stabilized zirconia

specimens displayed more consistent and flat surfaces than these groups, which justifies the R_a results.

Different surface treatments have been suggested to enhance the SBS between ceramic surfaces and orthodontic brackets (2,9,12-14,16,17), as debonding is frequently encountered (19). Nonetheless, the ideal surface treatment for bracket bonding is still unclear (9). Reynolds (21) reported SBS values higher than 6 MPa as clinically acceptable. However, SBS should also be at a reasonable level so that no cohesive damage occurs during debonding (10) and the least amount of adhesive remains on the zirconia surface (5). In this study, SBS values ranged between 2,5 MPa to 24,85 MPa for 4Y-PSZ, and from 2,42 MPa to 26,19 MPa for 5Y-PSZ. Furthermore, specimens of APA, MEP, and FS groups presented SBS values higher than 6 MPa. Therefore, these treatments may be considered clinically satisfactory.

Sandblasting produces a rough ceramic surface depending on the pressure and particle size (1,19). In this study, zirconia surfaces were treated with 50 µm Al_2O_3 particles at 0.2 MPa pressure. Several studies have evaluated the effects of sandblasting on the SBS of Y-PSZ and concluded that sandblasting with 0.2 MPa resulted in higher SBS values (2-4). However, a recent study reported similar values while comparing the SBS of metallic brackets to Er:YAG laser treated and sandblasted 5Y-PSZ. In addition, ARI scores of both groups were predominantly 2 (5). Contrarily, APA-5 had higher SBS values than ER-5 in the present study and this difference may be attributed to the parameters of the surface treatments. Furthermore, SBS of APA groups were higher than those of other groups, except for FS.

In a previous study, MEP treated zirconia was shown to have SBS higher than 6 MPa even after 10,000 cycles of thermocycling (13). This finding was further supported by another study, in which MEP provided SBS as high as 32.3 MPa after similar thermocycling (11). Similarly, SBS values of MEP groups exceeded 6 MPa, which may be associated with the significant increase in surface energy even with a minimal change in R_a (Figure 1, 2) (11). However, the knowledge on the effect of MEP on Y-PSZ is scarce and these results should be interpreted carefully. Furthermore, MEP has acidic components and possible toxic effects should be considered during intraoral application (18).

FSL irradiation of ceramics for orthodontic purposes has been scarcely studied (9,16,17). García-Sanz et al. (9) reported 200 mW output power and 60 µm inter-groove distance as the ideal parameters for treating 3Y-TZP. The same study also showed that these parameters led to higher SBS values than sandblasting (25 µm Al_2O_3 at a pressure of 2.5 bar for 20 s), which coincides with another study (16). In this study, FS resulted in nonsignificantly higher SBS values than APA, which may be associated with different parameters used. Although adequate SBS was achieved, clinical utilization of FSL is questionable considering the system costs and dimensions (16).

Specimens of APA and FS groups mainly had ARI score 3, which is an indicator of bond failure between orthodontic cement and metallic bracket. Considering that the greater amount of cement retained on the restoration surface means less chance of ceramic damage, FSL irradiation and sandblasting may be considered as the most zirconia-friendly treatments. However, this study did not evaluate the possible effects of these treatments on the mechanical behavior or phase transformation of Y-PSZ. Therefore, future studies investigating these parameters are needed to support this interpretation.

Even though the present study aimed to compare new-generation zirconias, absence of 3Y-TZP, which can be used monolithically in the posterior region is a limitation. In addition, the present study did not involve a zirconia primer, which may increase SBS (7). Another limitation was that a checkered depression pattern was created for FS groups. However, it is possible to engrave other geometrical designs (9,16) and distinct patterns might affect R_a and SBS. Since the esthetic expectations of patients are rising, preference of metallic bracket might also be a limitation.

Conclusion

Within the limitations of this study, the type of Y-PSZ did not affect R_a or SBS values. FSL irradiation and sandblasting emerged as the most effective and zirconia-friendly treatments. Single-step self-etching primer may be a valid surface treatment for Y-PSZ.

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Ethics

Ethics Committee Approval: This article does not contain any studies with human participants or animals performed by any of the authors.

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Authorship Contributions

Surgical and Medical Practices: M.D., Y.G., Concept: M.B.D., B.B.D., Design: M.B.D., B.B.D., Data Collection or Processing: B.B.D., M.D., Analysis or Interpretation: M.B.D., Y.G., H.Ş.K., Literature Search: B.B.D., M.D., Writing: M.B.D., Y.G., H.Ş.K.

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