ORIGINAL ARTICLE

Surface roughness and morphologic changes of zirconia: Effect of different surface treatment

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Abstract

Purpose: The purpose of this study was to investigate the surface roughness and morphologic changes of pre-sintered ZrO₂ after sandblasting and erbium, chromium: Yttrium, scandium, gallium, garnet (Er, Cr: YSGG) laser application of different intensities.

Material and Methods: Eighty pre-sintered ZrO_2 cylinders (7 mm diameter, 3 mm height) were prepared and divided into eight groups. Specimens in the control group were not treated. The following treatments were applied: Er, Cr: YSGG laser irradiation with different energy intensities (1-6 W at 20 Hz, with air-water cooling proportion of 65%/55%) and air abrasion with Al₂O₃ particles (120 µm). Then, all the specimens were sintered. The average surface roughness of each specimen was determined with a profilometer, and the morphology changes of a specimen from each group were evaluated with scanning electron microscope (SEM) analyses. The surface roughness data were analyzed through one-way analysis of variance and Tukey's honestly significant difference test (P < 0.05).

Results: There were significant differences between 2 and 6 W irradiations and control group. The highest surface roughness value was obtained with 6 W irradiation (8.14 ± 1.26 Ra), followed by the 5 W (7.60 ± 1.12 Ra), 4 W (7.50 ± 0.90 Ra), 3 W (5.86 ± 1.03 Ra), 2 W (4.54 ± 0.53 Ra) and sandblasting group (2.18 ± 0.92 Ra). 1 W laser irradiation (0.80 ± 0.06 Ra) presented Ra values similar to the control group (0.77 ± 0.03).

Conclusion: The result of the statistical analyses and SEM images showed that Er, Cr: YSGG laser irradiation with 4-6 W/20 Hz presented significantly effect in surface roughness changes of zirconia than other surface treatments.

Key words: Erbium, chromium: Yttrium, scandium, gallium, garnet laser, scanning electron microscopic, surface roughness, Y-TZP zirconia

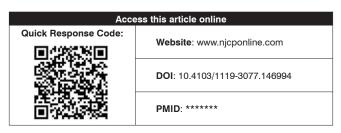
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Introduction

Nowadays, zirconia is the most popular dental material for patients and dentists because of their superior mechanical properties, such as high flexural strength (700-1200 MPa), fracture toughness (7-10 MPa m^{1/2}), high biocompatibility and natural appearance.^[1-3] Hence, zirconia ceramic material has a wide clinical usage, especially including implant abutments,^[4] and frameworks for fixed restorations.^[5-7]

An effective bonding relies on micromechanical interlocking and adhesive chemical bonding between zirconia and the

Address for correspondence: Dr. O Kirmali, Department of Prosthodontics, Faculty of Dentistry, Akdeniz University, Antalya, Turkey. E-mail: omerkrml@ymail.com resin cement or zirconia, and veneer ceramic is the most important factor for the long-term success of zirconia restorations. Besides, obtaining a desirable adhesion between ZrO_2 surface and cement or ZrO_2 surface and veneering porcelain requires surface pretreatment to improve the retention and fracture resistance of restorations.^[8-11] Previous investigations have been focused on different surface treatment for improving the bonding potential,^[12-14] increasing the surface area, creating a stronger micromechanical interlock.^[15,16]



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Researchers evaluated the effect of the aggressive mechanical abrasion methods used to increase surface roughness on ZrO_2 . These treatments are: Abrasion with diamond (or other) rotary instruments,^[17] air abrasion with alumina (or other) particles (Al_2O_3),^[14,18,11] grinding,^[19] acid etching (typically HF),^[11] laser^[9,10,11,14,20-27] and a combination of any of these techniques. However, acid etching application is not suitable for ZrO_2 because it does not have a glassy phase.^[14,28]

Erbium: Yttrium-aluminum garnet (Er: YAG) laser ($\lambda = 2.940$ nm) and neodymium: Yttrium aluminum-garnet (Nd: YAG) laser ($\lambda = 1.064$ nm) especially were used for surface treatment on ZrO₂ for obtaining the best bonding strength, and researchers reported that both of these lasers can be used effectively for changing the morphological characteristics of ZrO₂.^[9,10,14,21,22]

The erbium, chromium: Yttrium, scandium, gallium, garnet (Er, Cr: YSGG) laser has been introduced in dental clinics to remove carious dental hard tissues and to evaluate the morphological changes in human enamel and dentin that have been irradiated by it. However, recently some studies have evaluated the effects of the Er, Cr: YSGG laser irradiated on the shear bond strength of resin cement to ceramic restorations.^[24,25] However, a literature investigation showed that no study was found that evaluated the effect of Er, Cr: YSGG laser irradiation on ZrO₂.

Therefore, the aim of this study was to evaluate the effect of Er, Cr: YSGG laser irradiation of different intensities and air abrasion treatment on presintered ZrO_2 . The null hypothesis was that Er, Cr: YSGG laser irradiation of different intensities will change presintered ZrO_2 surface roughness and morphology.

Materials and Methods

Eighty pre-sintered ZrO_2 cylinders (Noritake Co, Nagoya, Japan) (7 mm diameter, 3 mm height) have been selected for this study. Specimens were sanded with 600-, 800-, and 1200-grit silicon carbide abrasives (English Abrasives, London, England) by a sander machine (Phoenix Beta Grinder/Polisher, Buehler, Germany) under water for 15 s and at 300 rev/min to be able to create a standard surface, and were randomly divided into eight groups (n = 10) according to the surface treatments performed:

- 1. Control: Specimens in the control group were not treated
- 2. Laser irradiations: All the surface of specimens was subjected to Er, Cr: YSGG laser irradiation (Millenium; Biolase Technology, Inc., San Clemente, CA, USA) with a 2.78 μ m wavelength, pulse duration from 140 to 200 μ s with a repetition rate of 20 Hz, the output power of this equipment ranges from 0.25 to 6.0 W. The optical fiber of the laser (600 μ m diameter, 6 mm length) was placed perpendicularly to the surface at 10 mm distance and was

moved in a sweeping fashion by hand during an exposure period of 20 s over the entire area. The energy parameters at 1 W, 2 W, 3 W, 4 W, 5 W, and 6 W, respectively, and water/air flow of 55% and 65%, respectively were used continuously during the irradiations

3. Sandblasting: The pre-sintered ZrO_2 surfaces were air abraded with 120-µm Al_2O_3 particles from a distance of 10 mm and at a pressure of two bars for 15 s.

Then, all ZrO_2 specimens were sintered at 1500°C for 8 h in a ZYrcomat (VITA Zahnfabrik, Sackingen, Germany) sintering furnace in accordance with the manufacturer's recommendation. The schematic test protocol used in the present study is shown in Figure 1.

Then, all specimens are ultrasonically cleaned for 3 min and specimens were stored in distilled water at 37°C for 24 h after the surface treatments.

All ZrO₂ specimens were mounted on metallic stubs, gold-sputter coated (Polaron Range SC 7620, Quorum Technology, Newhaven, UK), and evaluated for the morphological differences in the surface treatments applied on pre-sintered ZrO₂ surfaces with scanning electron microscope (SEM) (JSM-6060 LV, Jeol, Tokyo, Japan). Images from each group were taken at \times 5000 magnification. After the surface treatments, surface roughness (Ra, μ m) of each specimen was determined with a profilometer (Mitutoyo Surftest SJ-301, Japan) [Figure 2a]. The Ra value describes the average roughness value for a surface that was traced by the profilometer [Figure 2b]. Ten measurements at different locations were recorded for each specimen, and the average of these ten measurements was used to obtain the Ra value of each specimen. The surface roughness values were first checked for normal and equal distribution (Kolmogorov–Smirnov test, P = 0.01). The mean Ra values and standard deviations of the specimens were statistically evaluated parametrical analysis with one-way analysis of variance test in order to compare roughness values between different surface treatments, and multiple pair-wise comparisons were done with Tukey's honestly significant difference test (P < 0.05). The statistical analysis was handled with SPSS 15.0 (SPSS Inc., Chigaco, IL, USA).

Results

Table 1 presents mean and standard deviation values of the surface roughness (Ra, μ m) parameters for all groups. Results of statistical analyses indicated that there were significant differences among all groups. Comparison among the groups is shown in Table 1. All of the surface treatments tested produced rougher surfaces on the pre-sintered group. The surface irradiated at 6 W had the highest Ra, followed by the 5 W, 4 W, 3 W, and 2 W laser irradiated and air abrasion groups, respectively. In addition, the surface irradiated at 1 W and control surfaces showed the lowest values for the pre-sintered ZrO_2 specimens. Figure 3a and b shows SEM images of specimens with different surface treatments both pre

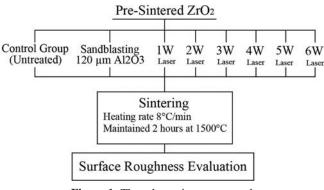


Figure 1: The schematic test protocol

sintered and after sintering. Similar to the laser irradiations, the air abrasion of the ZrO_2 surfaces showed morphologic

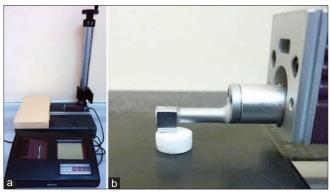


Figure 2: Evaluation of the surface roughness. (a) The photography of the profilometer, (b) measuring surface roughness of the pre-sintered ZrO₂

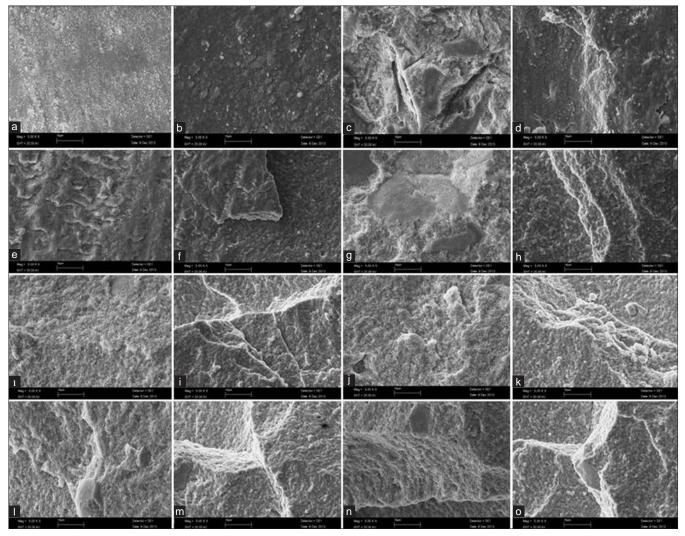


Figure 3: (a) Scanning electron microscopic images of different surface treatments on the surface of ZrO₂ (×5000) (a) No treatment presintered ZrO₂, (b) After sintering ZrO₂, (c) Sandblasted presintered ZrO₂, (d) After sintering ZrO₂ in letter "c", (e) 1 W laser irridiation presintered ZrO₂, (f) After sintering ZrO₂ in letter "e", (g) 2 W laser irridiation pre-sintered ZrO₂, (h) After sintering ZrO₂ in letter "g". (b) Scanning electron microscopic images of different surface treatments on the surface of ZrO₂ (×5000) (i) 3 W laser irridiation presintered ZrO₂, (i) After sintering ZrO₂ in letter "1", (j) 4 W laser irridiation presintered ZrO₂, (k) After sintering ZrO₂ in letter "j", (l) 5 W laser irridiation presintered ZrO₂, (m) After sintering ZrO₂ in letter "1", (n) 6 W laser irridiation presintered ZrO₂, (o) After sintering ZrO₂, in letter "n"

Table 1: Mean and SD value of the surface roughness (Ra, μ m)	
Surface treatment	Mean (SD)
Control	0.77 (0.03)ª
Air abrasion	2.18 (0.92) ^b
1 W laser	0.80 (0.06) ^a
2 W laser	4.54 (0.53)°
3 W laser	5.86 (1.03) ^d
4 W laser	7.50 (0.90) ^e
5 W laser	7.60 (1.12) ^e
6 W laser	8.14 (1.26) ^e

n=10, means with the same letters were not significantly different (P>0.05, Tukey's test). SD=Standard deviation

differences following different surface treatments. The specimen from control group had a typical untreated ZrO₂ surface [Figure 3a (a)]. More surface irregularities and deeper crevices were observed on pre-sintered ZrO₂ specimen from sandblasting group [Figure 3a-c] than on the control specimen [Figure 3a (a)]. The pre-sintered ZrO₂ specimen from laser groups also exhibited increased surface irregularity, as well as over destruction of the surface [Figures 3a and 3b]. Small pits, micro-cracks and irregularity were visible on pre-sintered ZrO, specimens from 1 to 6 W laser irradiations [Figure 3a (a-e, g) and b (i, j, l, n)]. In turn, more surface irregularities were observed in 6 W irradiation than in 4-5 W on pre sintered ZrO₂, despite the absence of a significant difference in surface roughness values [Table 1]. This roughness was observed after sintering with a tighter structure [Figure 3a and b].

Discussion

The surface roughness is important to obtain micromechanical retention for ZrO₂ ceramics. So, the researchers evaluated the effect of different surface treatments on the post sintered ZrO₂ to enhance the bonding strength with veneering porcelain or resin cement.^[22,26-32] But, some studies showed that post sintered surface treatments increase the fracture risk and damage ZrO, by increasing the content of the monoclinic phase.^[19,23,33-36] Guess et al.^[19] reported that post sintered surface treatment weakened the structure of ZrO₂ by causing micro-cracks. Similarly, Peterson et al.^[35] and Kosmac et al.^[36] reported that air abrasion treatment generated stress on the ZrO_2 surface and accelerated t-m transformation. Hence, Moon *et al.*^[33] investigated the effects of presintered surface treatments and found some advantages of this method. First, an effective roughness can be achieved on ZrO₂ surfaces, and secondly, it enhances the mechanical properties of ZrO, ceramics by increasing the content of the tetragonal phase.

The Er, Cr: YSGG laser, when used with an air-water spray, has been shown to cut enamel, dentin, cementum, and

bone efficiently and cleanly.^[24] The Er, Cr: YSGG laser has the ability to remove particles by a process called ablation, including micro-explosions and vaporization.^[11] On vaporization, the internal pressure builds within the tissue until the explosive destruction of the inorganic substance occurs before the melting point is reached.^[24]

In the present study, we aimed to investigate the effect of sandblasting and Er, Cr: YSGG laser irradiation with different energy intensities on the surface roughness of presintered ZrO_2 . According to the results of this study, the null hypothesis was accepted, as laser irradiations increased the surface roughness.

Air abrasion with Al₂O₃ particles, with sizes ranging from 25 to 250 μ m, is often done to provide undercuts, or to prepare a rough surface to constitute a strong adhesion of veneering ceramics or resin cement.^[29,33,34] Subasi and Inan^[27] evaluated the effect of different surface treatments on the surface roughness of ZrO₂, and found that all of the treatment methods tested increased the surface roughness values compared with untreated surfaces. And, they reported that air abrasion was the most effective surface treatment. Similarly, Demir et al.^[26] found the highest surface roughness value was obtained in the air abrasion group. Some previous studies examined the effects of different surface treatments on the surface roughness of ZrO₂ and found that sandblasting treatment increased the surface roughness values compared with untreated surfaces.^[23,26,27] In another study, Kirmali et al.^[9] reported that the values for shear bond strength of sandblasting of pre-sintered zirconia were statistically significant. Casucci et al.^[32] reported that sandblasting treatment significantly affected the roughness compared with untreated (7.31 Ra, 7.27 Ra, respectively) surfaces for Cercon (45.15 Ra) and Aadva Zr (51.67 Ra) ceramics.

Furthermore, Kirmali *et al.*^[23] examined the untreated, sandblasted, laser irradiations (Er: YAG and Nd: YAG laser) and combinations of these laser applications with sandblasting on presintered ZrO_2 and reported that the laser applications with sandblasting treatments and Er: YAG laser irradiation alone significantly increased the surface roughness values. In another study, Kirmali *et al.*^[9] found that Nd: YAG lasers decreased the shear bond strength compared to untreated and Er: YAG laser irradiation.

Cavalcanti *et al.*^[21] and Demir *et al.*^[26] examined the untreated, sandblasted, and Er: YAG laser application of different intensities on post sintered ZrO_2 surfaces, and Cavalcanti *et al.*^[21] reported that Er: YAG laser irradiation at 600 mJ significantly affected the surface roughness compared with the other groups. Demir *et al.*^[26] found that Er: YAG laser application of different intensities

increased the surface roughness, but the differences were not statistically significant. Besides, Miranda et al.^[37] evaluated the surface roughness on ZrO₂ surface after Er, Cr: YSGG laser irradiation at 1.5 W/20 Hz, air-water cooling proportion of 80%/25%, and found that laser irradiation decreased the surface roughness. This result is in accordance with Cavalcanti et al.[21], and did not coincide with the conclusions of Miranda et al.^[37] and Demir et al.^[26] For this reason, it may be thought that surface treatments were applied on a presintered ZrO, surface. However, Cavalcanti et al.[21] reported that higher laser power settings might cause heat damage to the ZrO, structure. Gökçe et al.[38] found similar results of different surface treatments to the ceramics. Also, Sari et al.^[39] evaluated the Er: YAG laser transmission ratio through different ceramics with different thicknesses and stated that the absorption of Er: YAG laser energy in ZrO, surface was quite low for to require surface modification.

Conclusions

Within the limitations of this study, it can be concluded that Er, Cr: YSGG laser irradiation with different energy intensities except 1 W, and air abrasion at 120 μ m Al₂O₃ represented effective methods for conditioning the ZrO₂ surface.

References

- Vagkopoulou T, Koutayas SO, Koidis P, Strub JR. Zirconia in dentistry: Part I. Discovering the nature of an upcoming bioceramic. Eur J Esthet Dent 2009;4:2-23.
- eHisbergues M, Vendeville S, Vendeville P. Zirconia: Established facts and perspectives for a biomaterial in dental implantology. J Biomed Mater Res B Appl Biomater 2009;88:519-29.
- Pittayachawan P, McDonald A, Young A, Knowles JC. Flexural strength, fatigue life, and stress-induced phase transformation study of Y-TZP dental ceramic. J Biomed Mater Res B Appl Biomater 2009;88:366-77.
- Brodbeck U.The ZiReal Post: A new ceramic implant abutment. J Esthet Restor Dent 2003;15:10-23.
- Raigrodski AJ, Chiche GJ, Potiket N, Hochstedler JL, Mohamed SE, Billiot S, et al. The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: A prospective clinical pilot study. J Prosthet Dent 2006;96:237-44.
- Vult von Steyern P, Carlson P, Nilner K. All-ceramic fixed partial dentures designed according to the DC-Zirkon technique. A 2-year clinical study. J Oral Rehabil 2005;32:180-7.
- Tinschert J, Schulze KA, Natt G, Latzke P, Heussen N, Spiekermann H. Clinical behavior of zirconia-based fixed partial dentures made of DC-Zirkon: 3-year results. Int J Prosthodont 2008;21:217-22.
- Kelly JR, Denry I. Stabilized zirconia as a structural ceramic: An overview. Dent Mater 2008;24:289-98.
- Kirmali O, Akin H, Ozdemir AK. Shear bond strength of veneering ceramic to zirconia core after different surface treatments. Photomed Laser Surg 2013;31:261-8.
- Akin H, Ozkurt Z, Kirmali O, Kazazoglu E, Ozdemir AK. Shear bond strength of resin cement to zirconia ceramic after aluminum oxide sandblasting and various laser treatments. Photomed Laser Surg 2011;29:797-802.
- Cavalcanti AN, Foxton RM, Watson TF, Oliveira MT, Giannini M, Marchi GM. Bond strength of resin cements to a zirconia ceramic with different surface treatments. Oper Dent 2009;34:280-7.

- Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Part II: Zirconia veneering ceramics. Dent Mater 2006;22:857-63.
- Della Bona A, Borba M, Benetti P, Cecchetti D. Effect of surface treatments on the bond strength of a zirconia-reinforced ceramic to composite resin. Braz Oral Res 2007;21:10-5.
- Spohr AM, Borges GA, Júnior LH, Mota EG, Oshima HM. Surface modification of In-Ceram Zirconia ceramic by Nd:YAG laser, Rocatec system, or aluminum oxide sandblasting and its bond strength to a resin cement. Photomed Laser Surg 2008;26:203-8.
- Kern M, Wegner SM. Bonding to zirconia ceramic: Adhesion methods and their durability. Dent Mater 1998;14:64-71.
- Senyilmaz DP, Palin WM, Shortall AC, Burke FJ. The effect of surface preparation and luting agent on bond strength to a zirconium-based ceramic. Oper Dent 2007;32:623-30.
- Sahafi A, Peutzfeldt A, Asmussen E, Gotfredsen K. Bond strength of resin cement to dentin and to surface-treated posts of titanium alloy, glass fiber, and zirconia. J Adhes Dent 2003;5:153-62.
- Xible AA, de Jesus Tavarez RR, de Araujo Cdos R, Bonachela WC. Effect of silica coating and silanization on flexural and composite-resin bond strengths of zirconia posts: An *in vitro* study. J Prosthet Dent 2006;95:224-9.
- Guess PC, Zhang Y, Kim JW, Rekow ED, Thompson VP. Damage and reliability of Y-TZP after cementation surface treatment. J Dent Res 2010;89:592-6.
- Blatz MB, Sadan A, Arch GH Jr, Lang BR. *In vitro* evaluation of long-term bonding of Procera AllCeram alumina restorations with a modified resin luting agent. J Prosthet Dent 2003;89:381-7.
- Cavalcanti AN, Pilecki P, Foxton RM, Watson TF, Oliveira MT, Gianinni M, et al. Evaluation of the surface roughness and morphologic features of Y-TZP ceramics after different surface treatments. Photomed Laser Surg 2009;27:473-9.
- da Silveira BL, Paglia A, Burnett LH, Shinkai RS, Eduardo Cde P, Spohr AM. Micro-tensile bond strength between a resin cement and an aluminous ceramic treated with Nd:YAG laser, Rocatec System, or aluminum oxide sandblasting. Photomed Laser Surg 2005;23:543-8.
- Kirmali O, Akin H, Kapdan A. Evaluation of the surface roughness of zirconia ceramics after different surface treatments. Acta Odontol Scand 2014;72:432-9.
- Usumez A, Aykent F. Bond strengths of porcelain laminate veneers to tooth surfaces prepared with acid and Er, Cr:YSGG laser etching. J Prosthet Dent 2003;90:24-30.
- Kursoglu P, Motro PF, Yurdaguven H. Shear bond strength of resin cement to an acid etched and a laser irradiated ceramic surface. J Adv Prosthodont 2013;5:98-103.
- Demir N, Subasi MG, Ozturk AN. Surface roughness and morphologic changes of zirconia following different surface treatments. Photomed Laser Surg 2012;30:339-45.
- Subasi MG, Inan O. Evaluation of the topographical surface changes and roughness of zirconia after different surface treatments. Lasers Med Sci 2012;27:735-42.
- Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: A review of the literature. J Prosthet Dent 2003;89:268-74.
- Curtis AR, Wright AJ, Fleming GJ. The influence of surface modification techniques on the performance of a Y-TZP dental ceramic. J Dent 2006;34:195-206.
- Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Effect of zirconia type on its bond strength with different veneer ceramics. J Prosthodont 2008;17:401-8.
- Kim HJ, Lim HP, Park YJ, Vang MS. Effect of zirconia surface treatments on the shear bond strength of veneering ceramic. J Prosthet Dent 2011;105:315-22.
- Casucci A, Osorio E, Osorio R, Monticelli F, Toledano M, Mazzitelli C, et al. Influence of different surface treatments on surface zirconia frameworks. J Dent 2009;37:891-7.
- 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;27:651-63.
- Monaco C, Cardelli P, Scotti R, Valandro LF. Pilot evaluation of four experimental conditioning treatments to improve the bond strength between resin cement and Y-TZP ceramic. J Prosthodont 2011;20:97-100.

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- Peterson IM, Pajares A, Lawn BR, Thompson VP, Rekow ED. Mechanical characterization of dental ceramics by hertzian contacts. J Dent Res 1998;77:589-602.
- 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;15:426-33.
- Miranda PV, Rodrigues JA, Blay A, Shibli JA, Cassoni A. Erratum to: Surface alterations of zirconia and titanium substrates after Er, Cr:YSGG irradiation. Lasers Med Sci 2014.
- 38. Gökçe B, Ozpinar B, Dündar M, Cömlekoglu E, Sen BH, Güngör MA. Bond

strengths of all-ceramics: Acid vs laser etching. Oper Dent 2007;32:173-8.

 Sari T, Tuncel I, Usumez A, Gutknecht N. Transmission of Er: YAG laser through different dental ceramics. Photomed Laser Surg 2014;32:37-41.

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