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 ZrO_2 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 Al_2O_3 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

Date of Acceptance: 25-Aug-2014

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 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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Access this article online

Quick Response Code:

Website: www.njcponline.com

DOI: 10.4103/1119-3077.146994

PMID: *******

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 (λ =2.940 nm) and neodymium: Yttrium aluminum-garnet (Nd: YAG) laser (λ =1.064 nm) especially were used for surface treatment on ZrO_2 for obtaining the best bonding strength, and researchers reported that both of these lasers can be used effectively for changing the morphological characteristics of ZrO_2.[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 ZrO_2.

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 μm wavelength, pulse duration from 140 to 200 μs 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 μm 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 ZrO_2 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 ZrO_2 surfaces with scanning electron microscope (SEM) (JSM-6060 LV, Jeol, Tokyo, Japan). Images from each group were taken at ×5000 magnification. After the surface treatments, surface roughness (Ra, μm) 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, μm) 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 sintered and after sintering. Similar to the laser irradiations, the air abrasion of the ZrO_2 surfaces showed morphologic

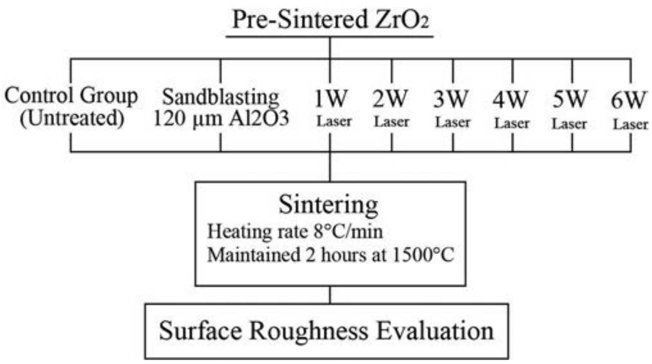

Figure 1: The schematic test protocol

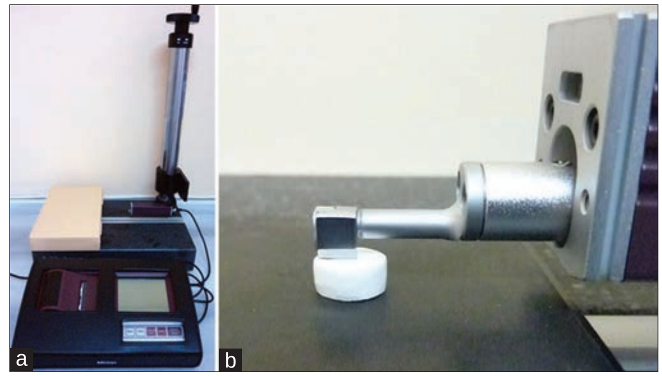

Figure 2: Evaluation of the surface roughness. (a) The photography of the profilometer, (b) measuring surface roughness of the pre-sintered ZrO_2

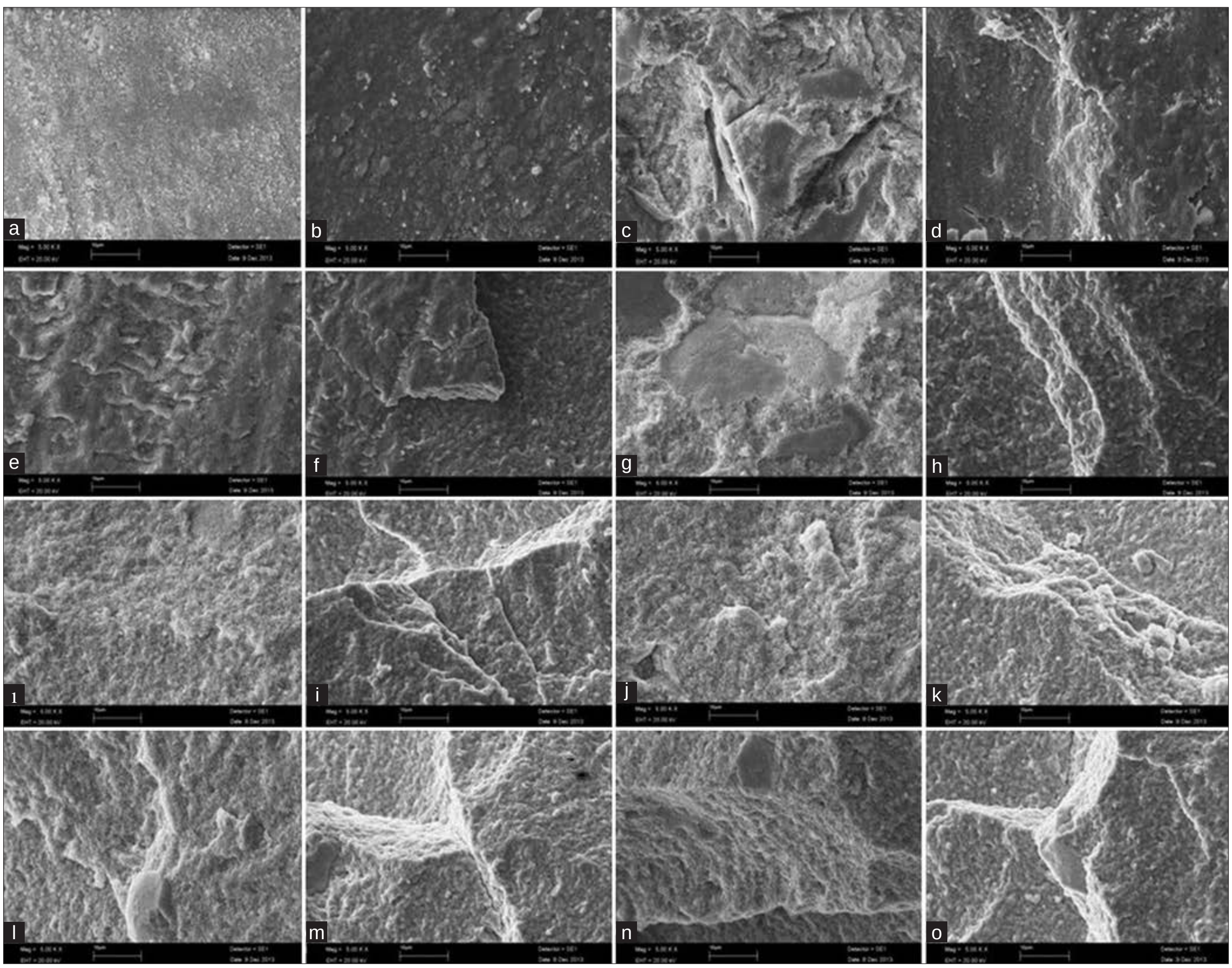

Figure 3: (a) Scanning electron microscopic images of different surface treatments on the surface of ZrO_2 (×5000) (a) No treatment presintered ZrO_2, (b) After sintering ZrO_2, (c) Sandblasted presintered ZrO_2, (d) After sintering ZrO_2 in letter "c", (e) 1 W laser irridiation presintered ZrO_2, (f) After sintering ZrO_2 in letter "e", (g) 2 W laser irridiation pre-sintered ZrO_2, (h) After sintering ZrO_2 in letter "g". (b) Scanning electron microscopic images of different surface treatments on the surface of ZrO_2 (×5000) (ı) 3 W laser irridiation presintered ZrO_2, (i) After sintering ZrO_2 in letter "ı", (j) 4 W laser irridiation presintered ZrO_2, (k) After sintering ZrO_2 in letter "j", (l) 5 W laser irridiation presintered ZrO_2, (m) After sintering ZrO_2 in letter "l", (n) 6 W laser irridiation presintered ZrO_2, (o) After sintering ZrO_2 in letter "n"

Table 1: Mean and SD value of the surface roughness (Ra, μm)

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)[c]
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 ZrO_2 surface [Figure 3a (a)]. More surface irregularities and deeper crevices were observed on pre-sintered ZrO_2 specimen from sandblasting group [Figure 3a-c] than on the control specimen [Figure 3a (a)]. The pre-sintered ZrO_2 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_2 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 ZrO_2, 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 ZrO_2 ceramics. So, the researchers evaluated the effect of different surface treatments on the post sintered ZrO_2 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_2 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 ZrO_2 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 ZrO_2 surfaces, and secondly, it enhances the mechanical properties of ZrO_2 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 Al_2O_3 particles, with sizes ranging from 25 to 250 μm, 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 ZrO_2, 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 ZrO_2 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 ZrO_2 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_2 surface. However, Cavalcanti *et al*.[21] reported that higher laser power settings might cause heat damage to the ZrO_2 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_2 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 μm Al_2O_3 represented effective methods for conditioning the ZrO_2 surface.

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How to cite this article: Kirmali O, Kustarci A, Kapdan A. Surface roughness and morphologic changes of zirconia: Effect of different surface treatment. Niger J Clin Pract 2015;18:124-9.

Source of Support: Nil, **Conflict of Interest:** None declared.