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 ZrO2 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 m1/2), 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 ZrO2 surface and cement or ZrO2 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]
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,\cite{17} air abrasion with alumina (or other) particles (Al$_2$O$_3$),\cite{14,18,11} grinding,\cite{19} acid etching (typically HF),\cite{18} laser\cite{9,10,14,20‑27} 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.\cite{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 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$_2$.\cite{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.\cite{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$_2$O$_3$ 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 ZrO₂ 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 ZrO₂ surfaces showed morphologic

![Figure 1: The schematic test protocol](image)

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

![Figure 3: 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 irradiation presintered ZrO₂, (f) After sintering ZrO₂ in letter “e”, (g) 2 W laser irradiation pre-sintered ZrO₂, (h) After sintering ZrO₂ in letter “g”, (i) Scanning electron microscopic images of different surface treatments on the surface of ZrO₂ (×5000) (i) 3 W laser irradiation presintered ZrO₂, (j) After sintering ZrO₂ in letter “i”, (k) 4 W laser irradiation presintered ZrO₂, (l) After sintering ZrO₂ in letter “j”, (m) 5 W laser irradiation presintered ZrO₂, (n) After sintering ZrO₂ in letter “k”, (o) After sintering ZrO₂ in letter “l”](image)
Table 1: Mean and SD value of the surface roughness (Ra, µm)

<table>
<thead>
<tr>
<th>Surface treatment</th>
<th>Mean (SD)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Control</td>
<td>0.77 (0.03)*</td>
</tr>
<tr>
<td>Air abrasion</td>
<td>2.18 (0.92)*</td>
</tr>
<tr>
<td>1 W laser</td>
<td>0.80 (0.06)*</td>
</tr>
<tr>
<td>2 W laser</td>
<td>4.54 (0.53)*</td>
</tr>
<tr>
<td>3 W laser</td>
<td>5.86 (1.03)*</td>
</tr>
<tr>
<td>4 W laser</td>
<td>7.50 (0.90)*</td>
</tr>
<tr>
<td>5 W laser</td>
<td>7.60 (1.12)*</td>
</tr>
<tr>
<td>6 W laser</td>
<td>8.14 (1.26)*</td>
</tr>
</tbody>
</table>

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

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 ZrO₂ by increasing the content of the monoclinic phase. [19,25,31-36] Guess et al. [19] reported that post sintered surface treatment weakened the structure of ZrO₂ by causing micro-cracks. Similarly, Peterson et al. [31] and Kosmac et al. [36] reported that air abrasion treatment generated stress on the ZrO₂ 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 ZrO₂ 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 ZrO₂. 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 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 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. [12,26,27]

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. [13] examined the untreated, sandblasted, laser irradiations (Er: YAG and Nd: YAG laser) and combinations of these laser applications with sandblasting on presintered ZrO₂ 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 ZrO₂ 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.\(^\text{[37]}\) 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.\(^\text{[21]}\), and did not coincide with the conclusions of Miranda et al.\(^\text{[37]}\) and Demir et al.\(^\text{[16]}\). For this reason, it may be thought that surface treatments were applied on a presintered ZrO\(_2\) surface. However, Cavalcanti et al.\(^\text{[21]}\) reported that higher laser power settings might cause heat damage to the ZrO\(_2\) structure. Gökteş et al.\(^\text{[18]}\) found similar results of different surface treatments to the ceramics. Also, Sari et al.\(^\text{[19]}\) 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 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\(_2\)O\(_3\) represented effective methods for conditioning the ZrO\(_2\) surface.

### References


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