

Surface modification of zirconia after laser irradiation

D. POPA, I.-R. BORDEA*, A. V. BURDE, B. CRIȘAN, R. S. CÂMPIAN, M. CONSTANTINIUC
University of Medicine and Pharmacy "Iuliu Hațieganu", Faculty of Dental Medicine, 32 Clinicilor Str., 400006, Cluj-Napoca, Romania

This study was conducted in order to identify zirconia surface modifications occurring after laser irradiation. Changes in roughness were investigated using three parameters: Ra in μm , Rz and Rq. Eighteen pieces of zirconia (square 15 mm board; 3 mm thickness) were prepared for the study. Specimens were randomly divided in two groups, each containing nine pieces. Group 1 was irradiated using neodymium: yttrium-aluminum-garnet (Nd:YAG) dental laser and Group 2 was irradiated using erbium: yttrium-aluminum-garnet (Er:YAG) dental laser. The specimens' surfaces were analyzed using a profilometer, before and after irradiation. The mean Ra values were computed from four profiles, for each group. The results were statistically analyzed by t-test at the 95% confidence level. There were no differences between the initial roughness of the specimens ($p=0.62$), but there were significant differences between Group 1 and Group 2 after irradiations ($p<0.001$). The results of the statistical analyses and macroscopic aspect showed that laser irradiation produces roughness changes of zirconia. Nd:YAG laser irradiation produced significantly higher alterations, in surface roughness of zirconia, than Er:YAG.

(Received July 4, 2016; accepted September 29, 2016)

Keywords: Dental CAD/CAM systems, Laser Therapy, Y-TZP zirconia, Roughness, Nd:YAG, Er:YAG

1. Introduction

Periodontal disease is one of the most common chronic infection in adults and very frequent in teenagers. Periodontitis is a bacterial-related inflammatory disease which leads to the destruction of tooth-supporting tissues. Successful periodontal treatment is dependent on the stoppage of tissue destruction, elimination or control of etiological agents together with a microbial shift toward one typically present in health [1, 2]. For treating periodontal diseases, the first nonsurgical step usually involves a special cleaning, called "scaling and root planing" to remove plaque and tartar deposits on the tooth and root surface. When periodontal pockets do not heal after scaling and root planing, surgery may be needed to better remove inflamed tissues and reduce the damage to the bone that has formed around the teeth [3]. In recent decades, considerable attention has been focused on the use of lasers (diode, erbium: yttrium-aluminum-garnet, neodymium: yttrium-aluminum-garnet, carbon dioxide, and erbium, chromium-doped yttrium-scandium-gallium-garnet lasers of varying wavelengths) as adjunct management approaches to enhance nonsurgical periodontal treatment, due to the fact that they offer a less invasive surgical approach [4, 5]. Treatment of tooth enamel with the ruby laser with a wavelength of 694nm showed a crater-like morphology surrounded by a glass-like appearance at the irradiated zone [6-11], which seems to be caused by fusing, melting and recrystallization of enamel crystallites. The exposure of human teeth to a ruby laser caused the surface enamel to be more resistant to in vitro subsurface demineralization [12].

Different laser types such as Nd:YAG, Er:YAG, Er,Cr:YSGG and CO₂ have been studied with respect to the effects they produce on the surface of dental materials [13].

A new class of dental materials for crown and fixed partial denture fabrication such as: high-aluminium trioxide (alumina) ceramics, leucite reinforced feldspathic ceramics, castable glass-ceramics, machining and CAD/CAM ceramic systems and yttrium tetragonal zirconia polycrystal (Y-TZP) has been introduced to the market [14].

The aim of this study was to evaluate the effect of Nd:YAG and Er:YAG laser irradiation on sintered ZrO₂. The null hypothesis was that Nd:YAG and Er:YAG laser irradiation will change sintered ZrO₂ surface roughness and morphology.

2. Materials and methods

A standard block of zirconia Z-CAD (Metoxit AG, Switzerland) was sliced, using a diamond disc cutter (Jota AG, Switzerland), into square pieces with 15mm board and 3mm thickness.

A total of 18 specimens were first cut and then sintered at 1350°C for 6 hours.

This ceramic consists of ZrO₂, Y₂O₃, HfO₂ and Al₂O₃ in different percentages (Fig. 1).

Chemical analysis / Chemische Analyse		
ZrO ₂ + HfO ₂ + Y ₂ O ₃	Weight / Gew. %	> 99.0
Y ₂ O ₃	Weight / Gew. %	4.5 – 6.0
HfO ₂	Weight / Gew. %	< 5.0
Al ₂ O ₃	Weight / Gew. %	< 0.5
Other oxides / Andere Oxide	Weight / Gew. %	< 0.5
Radioactivity / Radioaktivität	Bq/kg	<200
Chemical solubility / Chemische Löslichkeit	µg cm ⁻²	< 100
Physico-mechanical properties / Physikalisch-mechanische Eigenschaften		
Sintered density / Sinterdichte	g/cm ³	> 6.0
Flexural strength (3-point) / Biegefestigkeit (3-Punkt)	MPa	≈1200
Grain-size (mean linear intercept) Korngröße (MLI)	µm	< 0.4
Thermal expansion (25-500°C) / WAK (25-500°C)	10 ⁻⁶ /K	10.5
Fracture toughness K _{1c} / Bruchzähigkeit K _{1c}	MN/m ^{3/2}	8
Young's modulus / Elastizitätsmodul	GPa	210
Open porosity / Offene Porosität	%	0

Fig. 1. Chemical composition of Z-CAD Metoxit AG and physico-mechanical properties [16]

To ensure identical initial roughness, all specimens have one face from the original initial block, which was not touched with the cutter, called reference surface.

The specimens were divided into two groups, according to the performed surface treatment:

1. Nd: YAG laser irradiation (n=9): all reference surfaces of the specimens were Nd: YAG laser irradiated with a 1064nm wavelength, 4W, 20Hz. The optical fiber of the laser (300µm) was placed perpendicularly to the surface and was moved by hand during an exposure period of 60s over the entire area.

2. Er: YAG laser irradiation (n=9): all reference surfaces of the specimens were Er: YAG laser irradiated with a 2940nm wavelength, 2W, 50mJ, 40Hz.

Fotona Er: YAG laser optical fiber specifications: VARIAN 500/14, fiber tip efficiency: 90%, max. laser power: 2W, max. laser energy: 125mJ.

All sintered zirconia samples were analyzed using a surface roughness tester (Mitutoyo SJ-201, Japan) before and after the laser treatment. The analyzed parameters were: surface roughness (Ra, µm), ten-point height of irregularities (Rz=sum of the mean height of the five highest profile peaks and the mean depth of five deepest profile valleys measured from a line parallel to the mean line) and root-mean-square deviation of the profile (Rq= the square root of the arithmetic mean of the squares of profile deviation from the mean line [15]). The Ra values describe the arithmetic mean of the absolute values of the profile deviations from the mean line (Fig. 2)



Fig. 2. Evaluation of the surface roughness, measuring surface roughness of the sintered ZrO₂

The surface of the sample was imaginary divided into four pieces and four measurements, at different locations, were recorded for each sample. The average of these four measurements was used to obtain the Ra value of each specimen.

Statistical analysis was performed using MedCalc Statistical Software version 16.4.3 (MedCalc Software bvba, Ostend, Belgium; <https://www.medcalc.org>; 2016). Differences between repeated measurements were assessed by ANOVA for repeated measures, which took into account the type of laser used. A *p* value lower than 0.05 was considered statistically significant.

3. Results

Table 1 presents the results of the analyzed parameters: the mean values and standard deviation.

Table 1. Mean and SD value of the roughness (Ra, µm; Rz, Rq)

Surface treatment	RaMean (SD)	RzMean (SD)	RqMean (SD)
No treatment (before irradiation)	0.62 (0.50)	4.16 (0.47)	0.83 (0.06)
Group 1 (Nd:YAG)	1.08 (0.24)	5.75 (0.85)	1.38 (0.27)
Group 2 (Er:YAG)	0.68 (0.07)	4.64 (0.68)	0.92 (0.11)

N=9, SD=Standard deviation

The mean Ra values were computed from four profiles for each group. There were no differences between the initial roughness of the specimens (*p*=0.62) but there were significant differences between Group 1 and Group 2 after irradiations (*p*<0.001).

Fig. 3 and 4 shows macroscopic aspects of zirconia samples from Group 1 and Group 2.

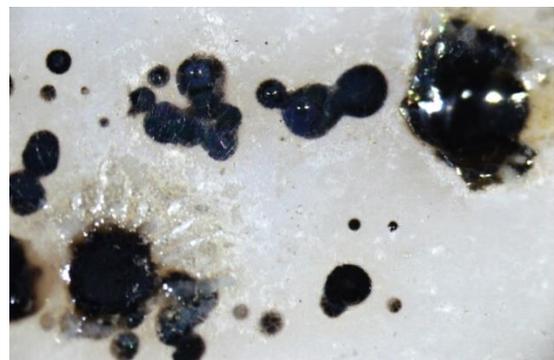


Fig. 3. Macroscopic aspect Group 1



Fig. 4. Macroscopic aspect Group 2

4. Discussions

More than twenty years ago, Metoxit AG was already one of the pioneers in the research and manufacturing of bioceramics. Over the years, Metoxit AG has expanded its know-how and now covers all steps of the production process from the raw powder to the final product. Whether axial or isostatic pressing – Metoxit always selects the production process which achieves the best final product [17].

Some studies reported that post sintered surface treatment weakened the structure of ZrO₂ by causing micro-cracks [18] and increases the fracture risk by incrementing the content of the monoclinic phase [18,19].

Studies of Cavalcanti et al. reported that Er: YAG laser irradiation at 400mJ or 600mJ produced considerable morphological changes and the surface roughness was significantly affected [20].

Spohr et al. showed that Nd:YAG laser treatment of In-Ceram Zirconia induced surface changes with material removal due to the micro-explosions, resulting in formation of voids, fusing and melting of the superficial layer. Nd:YAG laser irradiation of zirconia causes color change to black with many cracks and reduced oxygen content [21,22,23].

Our results are in agreement with these findings because the experimental zirconia samples showed roughness alteration, micro-explosions and black spots. The macroscopic aspect was more spectacular on Group 1 - irradiated with Nd:YAG, than Group 2 - irradiated with Er:YAG.

Laser technology may be used as a complementary technique as well as an alternative to traditional tools, adding many therapeutic advantages in restorative dentistry [24].

The clinician should be careful when applying lasers for periodontal disease on patients with zirconia restorations, because the surface of the restorative material can be affected, and even if the results are good for the moment, the roughness of the surface will retain, in short time, plaque and bacteria maintaining the inflammation of the soft tissues.

5. Conclusion

The results of the statistical analyses and macroscopic aspect showed that laser irradiation develop roughness changes of zirconia. Nd:YAG laser irradiation produced significantly higher alterations, in surface roughness of zirconia, than Er:YAG.

The irradiated surfaces showed an increased surface roughness compared to the non-irradiated surfaces.

Acknowledgments

This paper was published under the frame of European Social Found, Human Resources Development Operational Programme 2007-2013, project no. POSDRU 159/1.5/S/138776.

References

- [1] Marcus R. Karlsson, Christina I. Diogo Löfgren, Henrik M. Jansson. *Journal of Periodontology* **79**(11), 2021 (2008).
- [2] Walter Dukić, Ivona Bago, Andrej Aurer, Marija Roguljić, *Journal of Periodontology* **1** (2012).
- [3] American Dental Association. Available from: http://www.ada.org/~media/ADA/Publications/Files/patient_46.ashx
- [4] R. De Oliveira Guaré, S. C. Costa, F. Baeder, LA De Souza Merli, MT Dos Santos, A 2-year follow-up. *Spec Care Dentist* **30**, 46 (2010).
- [5] L. Palomo, J. M. Palomo, N. F. Bissada, *Semin Orthod.* **14**, 229 (2008).
- [6] L. Goldman, P. Hornby, R. Meyer, B. Goldman, *Nature* **4943**, 417 (1964).
- [7] R. H. Stern, R. F. Sognaes, *J. Dent. Res.* **43**(5), 873 (1964).
- [8] R. F. Sognaes, R. H. Stern, *J. S. Calif. Dent. Assoc.* **33**, 328 (1965).
- [9] R. Taylor, G. Shklar, F. Roeber, *Oral Surg.* **19**, 786 (1965).
- [10] R. R. Lobene, S. Fine, *J. Prosthet. Dent.* 1966; 16589.
- [11] J. Vahl, G. Pfefferkorn, *Dtsch Zahnärztl Z* **22**, 386 (1967).
- [12] R. H. Stern, R. F. Sognaes, F. Goodman, *J. Am. Dent. Assoc.* **73**, 838 (1966).
- [13] R. A. Convissar, E. E. Goldstein, *Dent. Today* **20**(1), 66 (2001).
- [14] Bülent Gökçe, Available from: <http://cdn.intechopen.com/pdfs-wm/30918.pdf>
- [15] Surface roughness tester. User's manual. Mitutoyo SJ-201, Japan.
- [16] Available from: http://www.metoxit.com/english/downloads/mat_z_cad_d_e.pdf
- [17] Available from: http://www.metoxit.com/english/downloads/dentalprosp_europa_e_web.pdf
- [18] P. C. Guess, Y. Zhang, J. W. Kim, E. D. Rekow,

- V. P. Thompson, *J. Dent. Res.* **89**, 592 (2010).
- [19] O. Kirmali, H. Akin, A. Kapdan, *Acta Odontol. Scand.* **72**, 432 (2014).
- [20] A. N. Cavalcanti, P. Pilecki, R. M. Foxton, T. F. Watson, M. T. Oliveira, M. Gianinni, et al. *Photomed. Laser Surg.* **27**, 473 (2009).
- [21] A. M. Spohr, G. A. Borges, L. H. Junior, E. G. Mota, H. M. Oshima, *Photomedicine and Laser Surgery* **26**(3), 203 (2008).
- [22] B. Gökçe, Available from:
<http://www.intechopen.com/books/scanning-electron-microscopy/effects-of-laser-irradiation-on-dental-hard-tissues-and-dental-materials-sem-evaluation->
- [23] M. Noda, Y. Okuda, J. Tsuruki, Y. Minesaki, Y. Takenouchi, S. Ban, *Dental Materials Journal* **29**(5), 536 (2010).
- [24] G. Olivi, M. Olivi, *Lasers in Restorative Dentistry. A Practical Guide*. Springer 2015.

*Corresponding author: Bordea.Ioana@umfluj.ro