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Original research

STRUCTURAL ALTERATIONS OF ZIRCONIA DEPENDING ON SINTERING PARAMETERS AND EFFECTS ON BOND STRENGTH AFTER DIFFERENT SURFACE TREATMENTS

*Mehmet Emre COŞKUN[1]
Fehim ÇELENK[2]

ORCID IDs of the authors:
M.E.C. 0000-0002-2430-5170
F.Ç. 0000-0002-7484-2855

[1] Department of Prosthodontics, Faculty of Dentistry, Cumhuriyet University, Sivas, Turkey
[2] Division of Prosthodontics, Alanya Public Health Center, Antalya, Turkey.

ABSTRACT

Objectives: Zirconia having different physical and optical properties is obtained after the application of different sintering parameters. This study aims to investigate structural variations after administering different sintering protocols and to evaluate the effect of different surface treatments on shear bond strength.

Materials and Methods: Eighty translucent zirconia specimens (7x3 mm) were divided into two different sintering groups (1480 ^{0}C for 180 min; 1510 ^{0}C for 30 min), then divided into four subgroups according to surface treatments: control, sandblasted, Er-YAG, and Nd-YAG (n:10). One specimen from each group was analyzed with XRD and one from each subgroup was analyzed with SEM. Ceramics (5x3 mm) were fired onto the zirconia for shear bond strength test using universal testing machine and the failure mode was determined by using stereomicroscope. Translucency and contrast ratio were measured by using spectrophotometer, and biaxial flexural strength test performed by employing universal testing machine on specimens with a diameter (15x1,3 mm) from each sintering group (n:10). Data was analyzed by using two-way ANOVA and Bonferroni Post hoc tests ($p<.05$).

Results: The short sintering group showed higher biaxial flexural strength (943.87±48.69 MPa). The highest surface roughness values were obtained in short sintering groups and within the groups Nd-YAG application was found the most effective method (4.11±0.28 Ra). The highest bond strength value was obtained in sandblasted short time sintered group (29.71±2.52 MPa). The translucency and contrast ratio showed no significant difference.

Conclusions: Although a physically stronger zirconia is obtained by short sintering process, long-term sintered zirconia forms a more durable bond strength with ceramics. Sandblasting improve the ceramic-zirconia bond strength may have more benefits than the use of Er-YAG, and Nd-YAG lasers.

Keywords: Shear bond strength, sintering, surface treatment, zirconia.

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***Corresponding Author:**
Sivas Cumhuriyet University, Faculty of Dentistry, Department of Prosthodontics, Turkey.
Phone: +90 346 219 10 10 ext 2758 **Fax:** +90 346 219 12 37 **Email:** dtcoskun@hotmail.com

INTRODUCTION

Despite the long-term success of metal-ceramic restorations, high aesthetic expectations of patients have increased the demand for full-ceramic restorations. Zirconia, among the full-ceramic restoration materials, has gained the popularity in prosthetic rehabilitation, due to the superior mechanical properties; high flexural strength (700-1200 MPa) and fracture toughness (7-10 MPa $m^{1/2}$).[1-4] However, the main problem of using zirconia is optical, since it is not as translucent as natural tooth structure or glass ceramics.[5] Two different proceedings have been carried out to overcome the esthetic problem of zirconia. The first one is using the zirconia as a framework and veneered with feldspathic ceramic. However, using the layered zirconia, it has been stated that chipping problem of the veneering ceramic is 15.2% for the posterior restorations after five-years usage.[6] The latter method is to make a full anatomical monolithic restoration, yet these restorations are aesthetically unsatisfactory when compared to the anterior teeth because of the opacity. Whichever technique is chosen to fabricate a prosthetic restoration that can mimic the natural teeth appearance to achieve better esthetic results, the main problem to be solved is making the zirconia more translucent.

In order to compensate the opacity of zirconia, sintering parameters have become a major interest in dentistry especially after the invention of state of art processing furnace that shorten the sintering cycles. The applied maximum temperature and dwelling time plays a decisive role on grain size, translucency, surface roughness (SR), porosity, and density.[4,7,8]

The grain size of the zirconia that effects the physical and optical properties, increases with the increasing temperature and dwelling time.[9-11] The increased grain size improves the translucency[12,13] however, expansion of the grain size deteriorates the mechanical strength and it was reported that the sintering exceed 1550 ^{0}C decreased the flexural strength remarkably.[9] Furthermore, the increasing grain size as a result of higher sintering temperature and dwelling time has a reducing effect in surface roughness (SR). The importance of enhanced SR has been emphasized in literature for providing mechanical retention to get adequate bond strength between ceramic and zirconia.[14] In literature different surface treatment methods such as sandblasting, acid etching, and laser applications (Er:YAG, Nd:YAG, CO_2, femtosecond) have been applied to improve the bond strength.[15-24] It' s often to perform the Er:YAG laser on cavity preparation and ceramic surface treatments because of its wavelength consistency with water.[17] Furthermore, it is mentioned that the Nd:YAG laser is an effective method in order to improve the resin-ceramic bond strength.[18]

Most of the studies in the literature aim to determine the effect of different sintering parameters on optical and physical properties of the material. However, there is limited study on the effect of altering physical properties on shear bond strength (SBS) between zirconia and ceramic. So, this study aimed to investigate the effect of different sintering parameters on the optical properties in terms of translucency parameter (TP), contrast ratio (CR) and as well as mechanical properties such as fracture resistance, surface roughness (SR), and grain growth of zirconium. Further, the study evaluated the effect of mechanical properties altered by different sintering protocols on SBS between zirconia and ceramic after the application of different surface treatment modalities. The hypothesis was that the high sintering temperature would enhance the mechanical and optical properties but not influence the bond strength.

MATERIAL AND METHODS

Specimens were prepared from partially sintered state zirconia (Optimadent; Upcera, China) using Computer-aided design (CAD) (DWOS; Dental Wings, Canada) and computer-aided manufacturing (CAM) (D40; Yenadent, Turkey). Specimens' surfaces were polished using P600, P800, and P1200 grid silicon carbide paper (English abrasives; Atlas, Turkey) sequentially. All specimens were randomly divided into two groups according to the sintering protocols (n:40). Long sintering (Ls) protocol was performed at 1480 ^{0}C for 180 min (MOS 160/1; Protherm, Turkey) and short sintering (Ss) protocol was at 1510 ^{0}C for 30

min (inFire HTC Speed; Sirona Dental, USA). Each group was divided into four subgroups (n:10) according to the surface treatments as below:

Control group: No surface treatment was applied.

Sandblasting group: Samples surfaces were treated with 110 nm Al_2O_3 (Metoxide; Metoxide frits and chemicals, Germany) at 5 bar pressure for 15 sec from a distance of 10 mm using a sandblaster (Mikrotek; Turkey).

Nd-YAG group: Samples were irradiated with a Nd:YAG laser (λ= 1.064 nm) (Smarty A10; Deka, Italy). The following settings were used: 150 mJ of energy, 10 Hz frequency, 1.5 W power, and 2.94 μs pulse duration for 15 sec. The contact-type laser optical fiber was aligned perpendicular to the sample, maintaining contact between the fiber and specimens.

Er-YAG group: Surface irradiation were performed by using Er:YAG laser (λ =2.940 nm) (Smart 2940D Plus; Deka, Italy) in pulsed mode 2.94 μs for 15 sec with 1.5 W power, 10 Hz frequency, 150 mJ energy, and under water cooling (5 ml/min). The optical hand piece was held perpendicular to the surface at a distance of 10 mm.

Biaxial flexural strength tests

Piston-on-three ball technique was used in order to investigate the biaxial flexural strength. Three stainless steel balls with 3.2 mm diameter were placed in a 10 mm diameter circle at an angle of 120 degree on the surface and twenty specimens (diameter 15, thickness 1.3 mm) from each sintering group were placed onto these balls (n:10). Tests were performed with a universal testing machine (Lloyd LF Plus; Ametek, UK) with a 0.5 mm/min crosshead speed.

Characterization

The characterization of the zirconia specimens was performed using different techniques. One specimen (diameter 15, thickness 1.3 mm) from each sintering group was analyzed by x-ray diffraction (XRD) (AXS D8 Advance; Bruker, USA) to identify the crystalline phase with monochromatic CuKα. Then, the analyses were evaluated using a software program (Jade 6.0; Materials Data Inc). In addition, one specimen from each surface treatment group was analyzed using scanning electron microscopy (SEM) (LEO 440; Computer Controlled Digital, UK) for detailed characterization. Photomicrographs of every surface treated specimens were obtained at 50x, 2500x, 10000x magnification.

Surface roughness tests

After surface treatments, all specimens from all groups with the dimension (diameter 7 mm, thickness 3 mm) were cleaned ultrasonically with isopropanol for 10 min and then air dried (n:80). Roughened surfaces were measured with profilometer (Surftest SJ-301; Mitutoyo, Japan) 6 times and the average roughness values (Ra, μm) of the specimens were determined.

SBS tests

Zirconia specimens (diameter 7 mm, thickness 3 mm) were placed into the holes compatible with the specimens' dimension on the custom-made metal mold base part, and ceramic (Cerabien ZR; Kuraray Noritake Dental Inc, Japan) veneering application was performed onto zirconia specimens through the second layer of the mold having cylindrical holes (diameter 5, thickness 3 mm). Then, they were fired according to the manufacturer's instructions. All specimens were immersed in distilled water at 37 ^{0}C for 24 h and SBS tests were performed with a universal testing machine (Lloyd LF Plus; Ametek Company, UK) with a 1 mm/min crosshead speed. The fractured zirconia surfaces were analyzed by stereomicroscope (Stemi DV4; Carl Zeiss, Germany). The failure mode of the specimens was categorized as adhesive, cohesive, and mix.

Optical measurements

For optical measurements 10 specimens (diameter 10 mm, thickness 1 mm) were fabricated for each sintering group (n:10). Measurements were performed with a spectrophotometer (Easyshade advance; Vita Zahnfabrik, Germany) according to the CIELab* scale under the D65 light. Black background (CIE L*= 1.1 a*=13.8 b*=52.2) and white background (CIE L*= 17.6 a*= 2.0 b*= 6.6) were used in measurements. The measurements were performed three times for each sample. TP was calculated according to the formula $TP=[(L_B - L_W)^2 + (a_B - a_W)^2 + (b_B - b_W)^2]^{1/2}$, and the CR was calculated as Y_B / Y_W, where Y is $[(L+16) / 116]^3$ x

100, in both formulas B and W subscripts represent black and white backgrounds, respectively.[23]

Statistical analysis

Statistical analysis was performed with SPSS 22.0 software (SPSS; SPSS Inc, USA). Shapiro-Wilks test was used to assess the homogeneity of variance and the data showed normal distribution. Two-way ANOVA test followed by the Bonferroni Post hoc comparisons were performed with a significant difference ($p<.05$).

RESULTS

The identified crystalline phase of the zirconia specimens which were sintered by using different protocols was depicted in Figure 1.

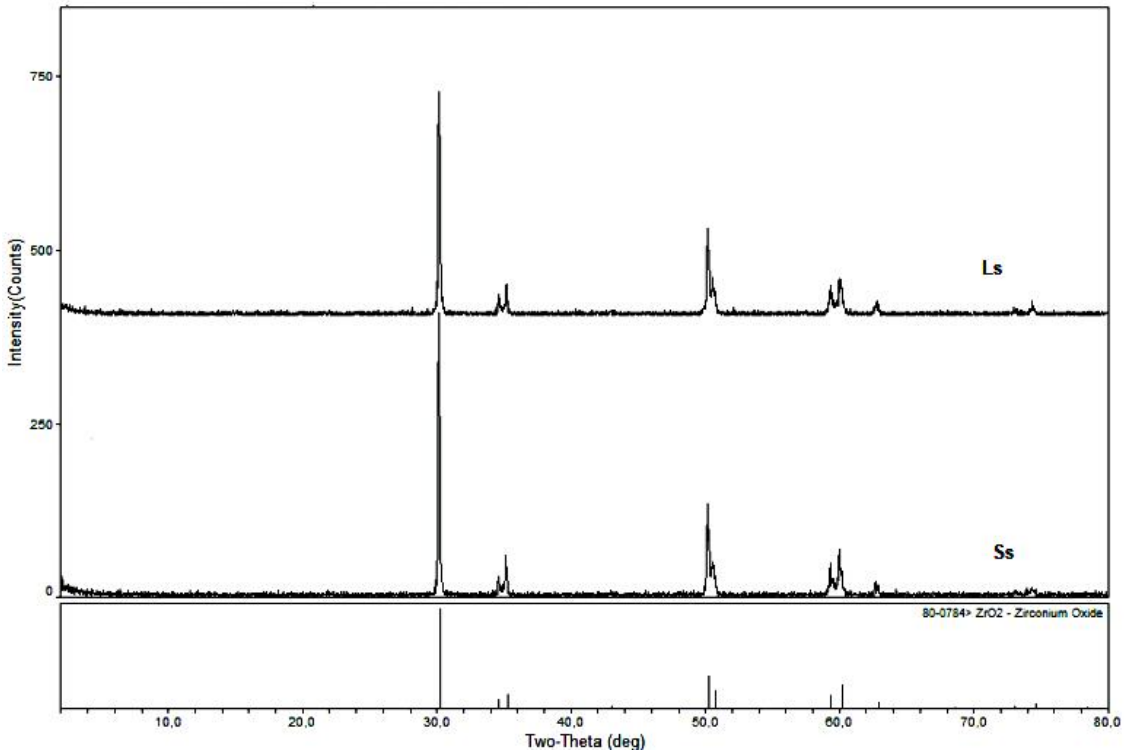

Figure 1. XRD analysis of the zirconia specimens

According to the XRD analyzes, the peak values for the spectra of both long and short sintered samples overlapped with 80-0784 ICSD tetragonal zirconia codes and besides short sintering group has larger grain size (1116 Å, 111 nm) than Ls group (555 Å, 55 nm)

The mean BFS of Ss group (943.872±48.69 MPa) was higher than the Ls group (779.651±52.34 MPa) and the difference between each group was statistically significant ($p<.05$) (Table 2).

Table 2. Mean values and standard deviations of TP, CR and BFS

	TP & Sd	CR & Sd	BFS & Sd (MPA)
Ss	2.63 ± 0.67^{a}	0.93 ± 0.016^{b}	943.87 ± 48.69^{x}
Ls	2.85 ± 0.42^{a}	0.92 ± 0.015^{b}	779.65 ± 52.34^{y}

* TP, translucency parameter; CR, contrast ratio; BFS, biaxial fracture strength. Means with same superscripts letters are not significantly different ($p>.05$).

Regarding the SR, all surface treatments increased the Ra values. The highest mean surface roughness was determined in Ss Nd:YAG group (4.11±0.28 Ra) and the lowest was measured Ss control group (0.83±0.29 Ra) (Table 1, Fig.2.).

Table 1. Surface roughness (Ra,µm), shear bond strength (MPa), biaxial flexural strength (MPa), standard deviations, and failure modes

	Surface Treatment	SR & Sd	SBS & Sd (MPa)	Mode of Failure		
				adhesive	cohesive	mix
Ss	**control**	0.83±0.29^{x}	20.82±1.40^{a,c}	8	0	2
	sandblasting	1.27±0.30^{y}	22.30±1.40^{a,b}	5	2	3
	Er-YAG	2.15±0.38	24.44±2.20^{b,d,e,f}	4	2	4
	Nd-YAG	4.11±0.28	26.54±1.88^{d,g,h,i}	1	3	6
Ls	**control**	0.90±0.23^{x}	24.23±1.58^{c,e,g,l}	8	1	1
	sandblasting	1.33±0.14^{y}	29.71±2.52^{k}	3	4	3
	Er-YAG	1.73±0.15	26.85±1.78^{f,h,l}	3	4	3
	Nd-YAG	3.17±0.14	27.92±2.04^{i,k}	2	4	4

*Ss, short sintering; Ls, long sintering; SR, surface roughness; Sd, standard deviation; SBS, shear bond strength. Means with same superscripts letters are not significantly different (p>.05)

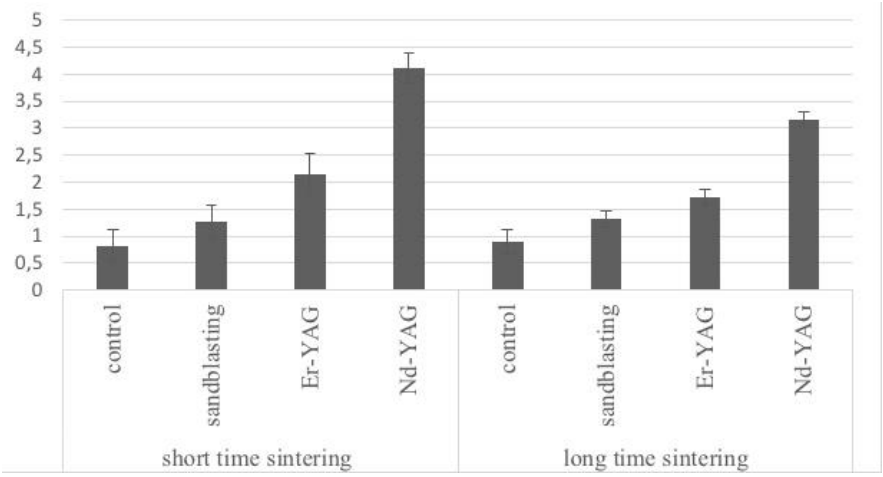

Figure 2. The mean and standard deviation of surface roughness (Ra)

The difference of surface roughness between all groups showed statistically significant. Only the differences between sandblasting groups and control groups showed no statistically significant difference. The SEM images of all zirconia samples showed in Figure 3.

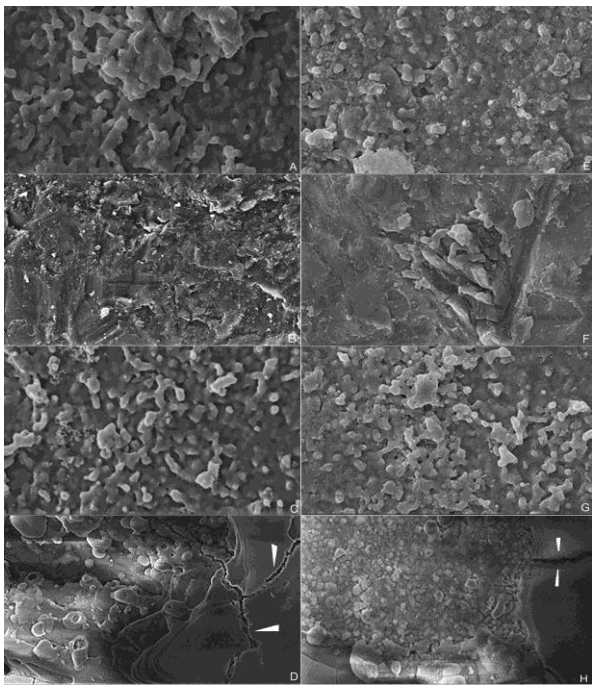

Figure 3. SEM images of zirconia specimens after different surface roughness and control groups: (A) Ls control, (B) Ls sandblasting, (C) Ls Er-YAG, (D) Ls Nd-YAG, (E) Ss control, (F) Ss sandblasting (10.00 K X); (G) Ss Er-YAG, (H) Ss Nd-YAG (2.50 K X). White lines in D and H figures show the crack regions on zirconia.

Concerning the shear bond strength, Ls groups showed higher bond strength than Ss groups (Table 1, Fig.4). The highest bond strength value was obtained in Ls sandblasting group (29.71±2.52) however the difference between Nd:YAG and sandblasting group was not statistically significant. In short sintering group, the highest bond strength value was obtained in Nd:YAG group, but no statistically significant difference was detected between Nd:YAG and Er:YAG group.

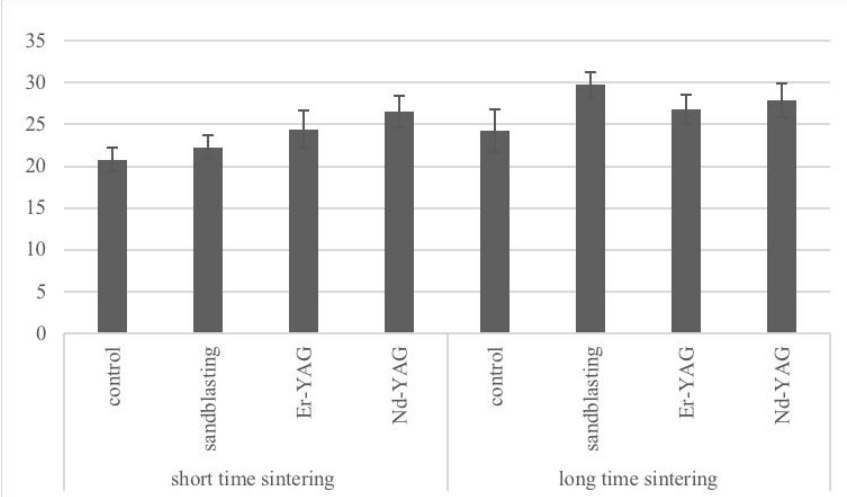

Figure 4. The mean and standard deviation of shear bond strength (MPa)

The TP and CR mean values were depicted in Table 2. Regardless of the sintering type, TP and CR values did not show statistically significant difference.

DISCUSSION

This study aimed to reveal the effects of different sintering processes on overall properties of zirconia. The sintering process is applied in order to get the desired microstructural properties of the zirconia such as particle size and pore distribution. In this study, the sintering parameters applied in the first group (Ls group) were designed in accordance with the manufacturer's instructions, while the second group (Ss) was planned as a shorter time application at higher temperature. The results obtained from this study showed that the increasing sintering temperature with a short application time enhance the mechanical properties while deteriorating the bond strength between ceramic and zirconia. The optical properties did not show any alterations due to the changing sintering conditions. Therefore, the tested hypothesis was partially rejected.

Grain size of the zirconia which is characterized by the sintering temperature and dwelling time, is the major determinant on optical and mechanical properties of the material[13]. The increasing temperature leads to the growth of the grains.[12] In the present study, two sintering protocols with different temperatures and dwelling times were tested on zirconia. In terms of grain size, higher molecular expansion was detected in Ss group, in which higher temperature and shorter dwelling time was applied. According to the results of this study, it was determined that the increasing dwelling time was not as effective as the temperature increase on the growth of grain size, and it may be summarized that the temperature was the main actor for characterization. Furthermore, Ebeid *et al.*[26] in his research mentioned that the granular expansion of zirconia occurred only after the 4 hours sintering at the same temperature. However, no dwelling time at that duration was applied in this research.

Concerning the optical properties of zirconia, Sen *et al.*[8] reported that the higher temperature increased the grain size which made TP higher. However, in the current study, no statistical difference regarding the TP and CR was found between sintering groups, while the grain size was different. Such a result may be attributed to the fact that the change in grain size was not sufficient to make a difference with regard to TP and CR.

Regarding the BFS, high temperature applied zirconia specimens (Ss group) showed a significant increase. The reason for the increased flexural

strength was the increase of grain size, which means reduction of the pores in zirconia's structure. Although this result is in agreement with Aydogdu, *et al*.[10] that aimed to reveal the effect of different sintering temperatures and dwelling times on biaxial flexural strength of zirconia, and mentioned that the temperature increase with a decreasing time enhanced the strength of the zirconia. However, in another research Ebeid, *et al.*[26] mentioned that the changing sintering parameters (time/temperature) did not alter the mechanical properties of zirconia. Furthermore, another study conducted by Stawarczyk, *et al.*[9] emphasized that the temperature increase up to 1550 had favorable effect on mechanical properties of zirconia, besides the exceed of this temperature decreased the fractural strength. So, there is an inconsistency among the studies however, it is obvious that the higher sintering temperature leads to the expansion of grain size. For this reason, to obtain a higher strength with the increasing temperature seems logical.

The changes in the surface texture of the zirconia after sintering process are clearly indicated in many researches[26,27] in the literature. In sintering process, the initial heating is occurred from the surface than in time, varies depending on the thickness, penetrate to the inner portion of the material and increase the grain size whereby the pores between grains decreased. In the case of applying the same temperature, the factor that determines the surface topography is the dwelling time, and vice versa. However, in this study both time and temperature were different and within the limitations of this study no significant difference was obtained because of the small temperature difference between the groups.

It is injudicious not to expect that all the changes of physical properties of zirconia have no effect on zirconia-ceramic bond strength. The final physical properties of zirconia effect the effectiveness of the surface treatments. Each type of laser, a form of energy, was found to be proper in order to get a high surface roughness as Er-YAG and Nd-YAG roughen the surface higher than that of the sandblasting and control group in both sintering groups (Table 2). Sandblasting, the mechanical surface treatment method, was not be able to form a rough surface (Ra, μm) like laser treatments. Although in literature it was shown that the laser applications improve the surface roughness[15,18], there is no consensus on the comparison of the effect of different surface treatments on ceramic-zirconia bond strength. Several studies[19,20] in the literature indicated that the sandblasting was more effective in order to increase SBS between ceramic and zirconia however, Korkmaz, *et al.*[21] emphasized that the sandblasting had no effect on bond strength, besides Kirmali, *et al.*[22] obtained the mean in vitro SBS of veneering ceramic on sandblasted zirconia only higher than the control group. In all studies only one sintering protocol was used which determine the final physical properties of the material and the difference of this research from the mentioned studies is the testing the different surface treatment on differently sintered zirconia.

In the current study, both type of laser application found to be the appropriate method in order to change the surface topography despite the diversity of physical properties of the zirconia determined by sintering. However, the results of this study indicated that, the same surface treatment application had different effect on Ss and Ls groups. The difference in hardness of the zirconia samples after the sintering process had an effect on the effectiveness of the surface treatments applied on them. In the case of the samples having a harder structure (Ss group), the laser efficiency was found to be higher and the mechanical method, sandblasting, was made a little difference which was not significant between Ls and Ss groups. Besides, the Nd:YAG laser will be more useful than Er:YAG laser for surface roughening of zirconia. It should be pointed out that the Nd:YAG ablation caused destructive effect on the zirconia surface due to the high temperature gradient which caused phase transformation ($t \rightarrow m$).[17] High temperature increases during the Nd:YAG application melted the zirconia surface and then cracks (in Fig. 2 G and H between white lines) were formed after sudden cooling that deteriorated the bond strength of ceramic-zirconia. In literature some studies mentioned the formation of microcracks on zirconia after Nd:YAG

treatment[17,18] which is inconsistent with the results of this study. Another issue that needs to be mentioned is the formation of black carbonation zones on the surface of zirconia as a result of Nd:YAG application and this discoloration negatively affect the esthetic requirements.

Regarding the shear bond strength, the results of the current study showed that the bond strength was not directly proportional to the surface roughness and the shear bond values of all Ls groups were higher than the same treated Ss groups. In literature, several studies determined that the surface treatments like sandblasting[23,24] and laser applications[18] caused crystallographic transformation (t→m). The thermal expansion coefficient (TEC) of monolithic zirconia is 7.5 x 10^{-60}, tetragonal zirconia is 10.8 x 10^{-60} and that of the ceramic is 9.5 x 10^{-60} [19] The increase of the TEC difference between ceramic and zirconia decreased the SBS which shorten the life span of the zirconia restoration and that means the bond strength between ceramic and tetragonal zirconia is higher than the bond strength between ceramic and monolithic zirconia. Applying the higher temperature in sintering process increases the flexural strength and makes the zirconia harder. However, according to the recent study, easier phase transformation occurs when the zirconia becomes harder and deteriorates the bond strength of zirconia-ceramic. Furthermore, the crystallographic transformation as a result of laser applications on zirconia was higher than that of sandblasting.

The assessment of SBS can not be completed without the evaluation of failure mode. The mode of failure gives crucial and precise information about bonding effectiveness. Lower bond strength represented by adhesive failure and it is not preferred. Kim, *et al*.[20] and Fischer, *et al*.[23] declared the ineffectiveness of sandblasting on bond strength between ceramic and zirconia, and mentioned mix and cohesive failure modes on control groups. Furthermore, they emphasized that sandblasting caused the phase transformation that deceased the bond strength. On the contrary, in the light of the results of the current study, without any surface treatments the SBS value was the lowest and the failure mode was adhesive.

CONCLUSIONS

Initially, pre-sintered zirconia produced in the same process with the same content has the same properties, but after the application of different sintering processes, a material having different properties is obtained. Within the limitations of this laboratory research such as the difficulty of applying ceramic onto zirconia at the same diameter; it was concluded that;

The applied higher sintering temperature increased the strength of zirconia. Laser; form of energy applications caused phase transformation which deteriorate the shear bond strength between zirconia and ceramic. Sandblasting; mechanical surface treatment is the appropriate method in order to increase zirconia-ceramic bond strength.

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CONFLICT OF INTEREST STATEMENT

The authors report no conflict of interest.

Zirkonyanın Sinterleme Parametrelerine Bağlı Oluşan Yapısal Değişimleri ve Farklı Yüzey İşlemleri Sonrasında Bağlantı Üzerine Etkileri

Amaç: *Zirkonya farklı sinterleme parametreleri sonucunda farklı fiziksel ve optik özellikler kazanır. Bu çalışmanın amacı, farklı sinterleme protokollerinin zirkonyada oluşturduğu fiziksel değişiklikleri ve uygulanan farklı yüzey işlemlerinin bağlantı üzerine etkilerini araştırmaktır.* ***Gereç ve Yöntemler:*** *Seksen translusent zirkonya (7x3 mm) farklı sinterleme protokollerine göre (1480 ºC' de 180 dk; 1510 ºC' de 30 dk) 2 farklı gruba ayrıldı ve sonrasında uygulanan yüzey işlemlerine göre (kontrol, kumlama, Er-YAG ve Nd-YAG) 4 farklı alt gruba ayrıldı. Her gruptan birer örnek XRD incelemesine tabii tutulurken, her alt gruptan birer örneğin SEM analizi yapıldı. Bağlantı dayanım testi için zirkonya yüzeylerine seramik (5x3 mm) pişirildi ve üniversal test cihazında testler gerçekleştirildi, kırılma şekilleri steriomikroskop kullanılarak belirlendi. Biaxial bükülme direnci üniversal test cihazında 15x1,3 mm boyutlarındaki örneklerde gerçekleştirildi. Elde edilen veriler iki yönlü ANOVA ve Bonferroni Post hoc*

testleri kullanılarak değerlendirildi. (p<,05) ***Bulgular:*** *En yüksek bükülme direnci kısa süreli sinterleme grubunda tespit edilmiştir (943,87±48,69 MPa). Yüzey pürüzlülüğü bakımından en yüksek değerler kısa süreli sinterleme gruplarında bulunurken, gruplar içerisinde Nd-YAG uygulaması en etkili yöntem olarak bulunmuştur (4,11±0,28 Ra). Translusentlik ve kontrast oranları arasında önemli bir farklılık bulunmamıştır.* ***Sonuç:*** *Kısa süreli sinterleme işlemiyle fiziksel olarak daha kuvvetli bir zirkonya elde ediliyor olsa da uzun süreli sinterleme işlemiyle daha kararlı seramik bağlantısı elde edilmektedir. Kumlama işlemi seramik-zirkonya bağlantısını artırmada Er-YAG ve Nd-YAG uygulamalarına nazaran daha etkilidir.* ***Anahtar Kelimeler:*** *Zirkonya, sinterleme, makaslama bağlantı dayanımı, yüzey işlemi.*

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