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Effects of Different Surface Treatments on Flexural Strength of Zirconium Oxide Cores

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Research Article

History

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ABSTRACT

Objectives: The aim of this study was to investigate the effect of different surface treatments on the biaxial flexural strength of zirconia and to determine phase transformation before and after sintering.

Materials and Methods: 150 cylindrical specimens with the dimensions 15 mm diameter and 1,3 mm height were obtained from semi-sintered Y-TZP blocks. These specimens were randomly separated into subgroups; sandblasting, Er:YAG laser, Nd:YAG laser, Er:YAG laser+sandblasting, Nd:YAG laser+sandblasting, fine grain bur, coarse grain bur. Half of the semi-sintered Y-TZP samples were treated before sintering and the others were treated after the sintering procedures. No treatment was performed in control group. Biaxial flexural strength test was performed to all samples. X-ray diffraction analysis (XRD) were performed to identify transformed monoclinic phase. The data were analyzed by Kruskal-Wallis, Man Whitney U test and Wilcoxon test.

Results: Specimens that were treated before sintering had lower biaxial flexural strength. The highest biaxial flexural strength values in all groups were seen in sandblasting groups and the lowest in grinding groups. According to the XRD analysis the highest phase transforme was determined in sandblasting groups. Sandblasting, Er-YAG laser+sandblasting and Nd-YAG laser+sandblasting were greatly increased the biaxial flexural strength of all the surface treatments after sintering. All the sandblasting treatments were found more monoclinic phase was found than other groups.

Conclusions: Surface treatments were found to affect both the mechanical properties and phase changes of zirconia.

Key Words: Zirconia, Flexural strength, Phase transformation, Sandblasting, Er:YAG laser, Nd:YAG laser.

Zirkonyum Oksit Alt Yapı Üzerine Uygulanan Farklı Yüzey İşlemlerinin Bükülme Dayanımına Etkileri

Süreç

Geliş: 11/09/2023
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ÖZ

Amaç: Bu çalışmanın amacı, farklı yüzey işlemlerinin zirkonyanın iki eksenli bükülme dayanımı üzerindeki etkisini araştırmak ve sinterleme öncesi ve sonrası faz dönüşümüne etkilerini belirlemektir.

Gereç ve Yöntemler: Yarı sinterlenmiş Y-TZP bloklardan 15 mm çapında ve 1,3 mm yüksekliğinde 150 silindirik örnek elde edildi. Bu numuneler rastgele alt gruplara ayrıldı; Kumlama, Er:YAG lazer, Nd:YAG lazer, Er:YAG lazer+kumlama, Nd:YAG lazer+kumlama, ince grenli frez, kalın grenli frez. Yarı sinterlenmiş Y-TZP numunelerinin yarısına tam sinterleme yapılmadan önce, kalanlara ise tam sinterleme işlemi yapıldıktan sonra yüzey işlemlerine tabi tutuldu. Kontrol grubuna herhangi bir yüzey işlem uygulanmadı. Tüm örneklere iki eksenli bükülme dayanımı testi uygulandı. Monoklinik faz dönüşümünü tanımlamak için X-ışını kırınım analizi (XRD) yapıldı. Veriler Kruskal-Wallis, Man Whitney U testi ve Wilcoxon testi ile analiz edildi.

Bulgular: Tam sinterlemeden önce yüzey işlemi gören numuneler daha düşük iki eksenli bükülme dayanımı gösterdi. Tüm gruplarda en yüksek iki eksenli bükülme dayanımı değerleri kumlama gruplarında, en düşük değerler ise frezleme gruplarında görüldü. XRD analizine göre en yüksek faz dönüşümü kumlama grubunda görüldü. Tam sinterleme işlemlerinden sonra uygulanan kumlama, Er-YAG lazer+kumlama ve Nd-YAG lazer+kumlama yüzey işlemlerinin iki eksenli eğilme dayanımını büyük ölçüde artırdığı görüldü. Tüm kumlama işlemlerinde diğer gruplara göre daha fazla monoklinik faz değişimi görüldü.

Sonuç: Yüzey işlemlerinin zirkonyanın hem mekanik özelliklerini hem de faz değişimlerini etkilediği bulundu.

Anahtar Kelimeler: Zirkonyum Oksit, Bükülme Dayanımı, Faz dönüşümü, Kumlama, Er:YAG lazer, Nd:YAG lazer.

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Introduction

The use of zirconia ceramics in aesthetic dentistry has been on the rise in recent years. Zirconia ceramics are widely used in prosthetic restorations due to their advantages such as high mechanical properties, biocompatibility, good dimensional stability and color compatibility. The preferred core material for prosthetic restorations is Yttria-Stabilized Tetragonal Zirconia Polycrystals (Y-TZP).[1,2] While zirconia is found in nature in polymorphic form, it has monoclinic form (m), tetragonal form (t) and cubic form (c). The melting point of zirconium is 2680 ^{0}C and it is in the cubic phase up to this temperature. If it falls below this level, it transitions from cubic phase to tetragonal phase. The tetragonal phase is stable up to 2370 ^{0}C. When it is lowered below 2370 ^{0}C, the tetragonal structure turns into tetragonamonoclinic phase and this phase transition takes place below 1170 ^{0}C. When passing from the tetragonal phase to the monoclinic phase, the volume of the crystals increases (4%-5%), which causes the appearance of microcracks or macrocracks and the loss of their mechanical properties.[3,4] Zirconia is fragile at room temperature in the monoclinic phase. Therefore, in technical applications, stabilization of the compound is required to prevent the transition from the tetragonal phase to the monoclinic phase. This stabilization is carried out with Yttrium trioxide.[3,5]

As a result, the properties of zirconia can change not only depending on its content and microstructure, but also depending on the production method. Zirconia restorations can be produced from partially sintered blocks and then subjected to the final sintering process, as well as from fully sintered blocks.[6,7]

Improvement in the mechanical properties of zirconia is linked to its long-term performance. However, the clinical success of prosthetic restorations depends largely on cementation. Different surface treatments have been tried to obtain micro-retaining area on the zirconia surface and to increase the surface area. These surface treatments ensured the connection between zirconia and resins, and between zirconia and ceramics, and the successful use of the restoration for a long time.[8,9] These applications are sandblasting[10], grinding[11], laser[10-12] or a combination of these. Surface defects can also occur on zirconia materials during laboratory or chairside procedures.[2,8]

In occlusal adjustments made on zirconia after grinding, it was stated that there was a decrease in stress relief and flexural strength in zirconia in long-term follow-ups. It has also been reported that this decrease is related to the degree of conversion from the tetragonal phase to the monoclinic phase.[13] Although there are many studies on the effect of laser and sandblasting on bond strength[8,10,12], there are limited studies on the effect of laser treatment applied to zirconia on biaxial flexural strength.[14,15] Therefore, the aim of the present work is to investigate the effect of different surface treatments applied to zirconia before and after sintering on the flexural strength and phase transformation. The null hypotheses are that all surface treatments will not affect flexural strength and that surface treatments will not alter phase transformation.

Materials and Methods

Semi-sintered Y-TZP zirconia block (Noritake Dental Inc, Japan) was used in this study. The disc-shaped specimens were designed and milled using a CAD/CAM system (Yenamak, Yenadent Ltd., Istanbul, Turkey). A total of 150 disc-shaped specimens, 15mm in diameter and 1.3mm thick, were obtained from these blocks (n=10). After preparation, half of the samples were surface treated before sintering and sintered according to the manufacturer's instructions. In the remaining samples, surface treatments were applied after sintering. The sintering process was completed in a total of 8 hours by allowing it to come back to room temperature from room temperature to 1375 °C in the sintering furnace (Protherm Furnaces, Istanbul, Turkey). After all samples were sintered, surface treatment was applied to the untreated samples. In this way, two main groups were created. The groups are listed as follows

Control (C): Not surface treated

Sandblasting (S): Surfaces of the samples were sandblasted with 110 µm Al_2O_3 particles at pressure of 0.5 MPa for 15s and distance of 10mm (Blastmate II; Ney, Yucaipa, CA, USA). After which the samples were washed and dried

Er:YAG Laser (E): It was applied to the sample surfaces by scanning them for 20 seconds with an optical fiber transport system. The distance is adjusted to 10 mm. Er:YAG laser (Smart 2940D Deka Laser, Florence, Italy) was applied by adjusting the beam settings to 150 mJ, 1.5 W and 10 Hz.

Nd:YAG Laser (N): Nd:YAG laser (Smarty A10 Deka Laser, Florence, Italy) was applied to the sample surfaces from a distance of 10 mm from a distance of 10 mm for 20 seconds. Beam settings were set to 100 mJ, 1 W, 10 Hz.

Er:YAG Laser and Sandblasting (ES): First, the above Er:YAG laser parameter was applied in the same way. Then the samples were washed and dried. Afterwards the surface was sandblasted in the same parameter.

Nd:YAG Laser and Sandblasting (NS): First, the above Nd:YAG laser parameter was applied in the same way. Then the samples were washed in running water and dried, and then surface was sandblasted in the same parameter.

Grinding (Fine Grained Bur) (FG): Diamond burs with 50 µm grain size (Meisinger, Hansemannstr, Neuss, Germany) were preferred for grinding the samples. The bur was attached to the handpiece and the rotation speed per minute was set to 20000. At the end of the grinding process, the sample thickness was thinned by 0.1 mm. The thickness was measured with a digital caliper.

Grinding (Coarse Grained Bur) (CG): Diamond burs with a 200 µm grain size (Meisinger, Hansemannstr, Neuss, Germany) were used for grinding the specimens. The other operations were performed in the same way as with the fine-grained bur.

Biaxial flexural strength

A Universal machine (Lloyd Instruments, LF Plus Segensworth, Fareham, UK) was used for the biaxial flexure test according to ISO 6872.

Three balls with a diameter of 3.2 mm were placed on a 10 mm diameter circle. The balls were positioned at an angle of 120 degrees with respect to the center of the circle (Figure1). The sample was placed on the balls with its center on the same axis as the piston. Force was applied to the sample surfaces with a cylindrical tip with a diameter of 1.4 mm (Figure 2). The crosshead speed was set to 0.5 mm/min. The strength has been calculated in accordance with the formulas below:

$$S = - 0,2387\ P(X- Y)/d^2$$

S: Biaxial flexural strength (MPa), P: Force at break (N), d: Sample thickness (mm)

$$X = (1+ v) \ln(r_2/r_3)^2 + [(1-v)/2] (r_2/r_3)^2$$

$$Y = (1+ v) [1 + \ln(r_1/r_3)^2] + (1-v) (r_1/r_3)^2$$

v: Poisson' ratio (0.25), r_1: The radius of the circle on which the support balls are located (mm), r_2: Radius of the force applied field (mm), r_3: Radius of sample (mm).

X-ray diffraction analysis (XRD)

Crystal analyzes of the samples were performed with an XRD device (Bruker AXS D8 Advance, UK) using monochromatic CuKα heat. Scanning was performed on the sample surface between 20-40 degrees (2θ) with a 0.01 degree step interval. Intensity values found as a result of X-ray diffraction were recorded. In each of the samples, the highest value observed in the denser regions and the 2θ angles at which these values were observed were recorded. Amount (XM) of the phase-changed monoclinic phase on the field of the surface-treated samples compared to the tetragonal phase was calculated according to the equation stated by Garvie and Nicholson.[16]

$$X_M = \frac{I_{M(111)} + I_{M(11\bar{1})}}{I_{M(111)} + I_{M(11\bar{1})} + I_T}$$

I: The highest value of the phase density

M(111) : Plane showing (111) crystal geometry belonging to the monoclinic phase

M(111-) : Plane showing (111-) crystal geometry belonging to the monoclinic phase

T: Tetragonal phase

Statistical analyses

The data was uploaded to the SPSS (ver: 14.0) program. Analysis of Variance, Tukey's test and the significance test of the peer-to-peer difference were used in the evaluation of the data since the parametric test assumptions were fulfilled (p=0.05).

Results

The result of the biaxial flexural strength test applied to the test groups are explained in Table1. While S group showed the highest flexural strength among all groups, the lowest was seen in group FG and CG, respectively. (p=0.001). Before sintering surface treatment applications decreased flexural strength in all groups compared to after sintering surface treatment applications and it was found statistically significant in all groups except group FG (p=0122) and CG (p=0.106).

The results of the monoclinic phase content values (%) are demonstrated in Table 2. While the monoclinic phase content is seen between 1% and 2% in the groups that have been surface treated before sintering, it is seen between 1% and 13% in the surface treatments applied after sintering. The highest amount of the monoclinical phase was found Group S, ES and NS respectively. After the surface treatments after sintering, monoclinic peaks were seen with M (111) orientation in the XRD model (Figure 3).

Discussion

The effect of surface treatments on biaxial flexural strength and phase transformation before and after sintering was investigated in this study. According to the results, there was significant difference among the biaxial flexural strength and phase transformation all groups before and after sintering. The hypothesis that surface treatments would not affect the flexural strength and change the phase transformation of zirconia was rejected.

The mechanical and chemical surface treatments applied on the zirconia allow to increase the surface roughness and porosity and improve the wettability.[17] Thus, it affects the bonding of the ceramic to be applied on the zirconia. İn addition, It is necessary to know whether there is a change in the physical properties of these applied surface treatments other than bonding.

In the literature, different results can be seen on Y-TZP zirconia in the grinding process, which is one of the surface treatments. In some studies, grinding triggers the t-m phase change and creates compression stress with approximately 4% volumetric expansion at superficial defect sites and prevents crack propagation.[13,18] In addition, in other studies, it has been stated that grinding causes a decrease in its mechanical properties by creating catastrophic defects on zirconia.[19] In study, the decrease in flexural strength in surface treatment with burs of different grain sizes shows parallelism with the above study.

Sandblasting process are the parameters frequently used in surface treatments. Some authors indicated that sandblasting increase the flexural strength on zirconia and seemed to result from the increase in monoclinic phase content.[20-22] Caglar *et al.*[14] reported that 110 µm Al_2O_3 particles for 30 seconds on zirconia increased the monoclinic phase and flexural strength in all groups. In study, the surface treatments increase the monolithic phase content and the monoclinic phase content in the sandblasting processes shows the highest values in flexural strength, which supports the above study. In the grinding groups, it was observed that there was less monoclinic phase transforme, but a decrease in durability. It can be said that this may be due to the heat arising in the grinding application and the presence of microcracks on the surfaces. In other studies, it has been reported that various surface treatments result in different rates of phase transforme (t-m), but the flexural strengths are statistically similar.[21]

Laser has been used in dentistry since 1995. Many studies have been carried out to determine reliable values when using the ER:YAG laser on zirconia.[14,23,24] Cavalcanti *et al.*[23] reported that Er:YAG laser (200 mJ) was more trusted for zirconia ceramics between the 400 and 600 mJ densities.Akin *et al.*[24] reported that 150 mj Er:YAG laser increases the surface roughness. these days, we planned the laser energy release to be 150 mj.

Çağlar *et al.*[14] remarked that sandblasting showed higher flexural strength compared to the control group in different surface treatments on zirconia, and Er:YAG laser showed a similar but lower value compared to the control group. He stated that this result was achieved with the application of the laser with the water cooling process and the preservation of the monoclinic phase amount in its structure. They also stated that cracks on the zirconia surfaces in SEM examinations may be one of the reasons for reducing this strength. This result was similar to that of the present study, which reported that the relative amount of the monoclinic phase of Er:YAG laser treatments was close to that of zirconia control groups.

Kurtulmus *et al.*[15] reported that laser and sandblasting before sintering would reduce the flexural strength of zirconia. In study, all surface treatments before sintering illustrated lower flexural strength in zirconia compared to the surface treatments after sintering, and it was statistically significant between the groups. This result was similar to that of the present study.

Within the limitations of this study, it has been evaluated the surface treatments affect the flexural strength of zirconia. However, thermal aging process should be performed and its effect in the oral environment should be evaluated. In addition, it should be determined which one will be more effective by using different parameters in surface treatments. It is necessary to compare different parameters in determining the relationship between surface treatments and phase transforme.

Conclusions

- All sandblasting parameters increased the flexural strength of zirconia.
- All surface treatments before sintering significantly reduced the flexural strength of zirconia compared to after sintering.
- The surface treatment that the most reduced the flexural strength compared to the no surface teratment group was the grinding group.
- The most monoclinic phase transformation was seen with the sandblasting surface treatment.

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Table 1. Test results of biaxial flexural strength of all groups (MPa)

Groups	Before Sintering X ± Sd (MPa)	After Sintering X ± Sd (MPa)	
C Group	1171.72 ± 34.34a	1171.72 ± 34.34k	
S Group	1243.15 ± 29.14b	1287.41 ± 26,59l	t= 5.16 P= 0.001*
E Group	1000.45 ± 46.99c	1115.78 ± 22.91m	t= 7.15 P= 0.001*
N Group	1033.27 ± 53.15c	1101.31 ± 16.21m	t= 3.90 P= 0.004*
ES Group	1102.82 ± 36.38d	1229.43 ± 29.22n	t= 12.82 P= 0.001*
NS Group	1187.39 ± 30.60a	1232.15 ± 23.61n	t= 4.91 P= 0.001*
FG Group	937.11 ± 42.76e	976.23 ± 32.52o	t= 1.70 P= 0.122
CG Group	927.36 ± 27.18e	952.13 ± 32.46o	t= 1.79 P= 0.106
	F= 95.23 P= 0.001 P< 0.05	F= 181.91 P= 0.001 P< 0.05	

*When the mean values of each group before and after sintering are compared, the difference is statistically significant (p<0.05).
** The difference between the means followed with different lowercase letters in the vertical columns is statistically significant according to the Tukey test (P<0.05).

Table 2. Relative amount of monoclinic zirconia (%)

Gruplar	Before sintering	After sintering
C Group	1.46	1.46
S Group	2.02	13.4
E Group	1.94	3.46
N Group	1.82	2.13
ES Group	1.99	11
NS Group	1.71	10.76
FG Group	1.62	6.62
CG Group	1.73	8.86

Figure 1. Positioning of stainless steel balls

Figure 2. The sample was placed on stainless steel balls.

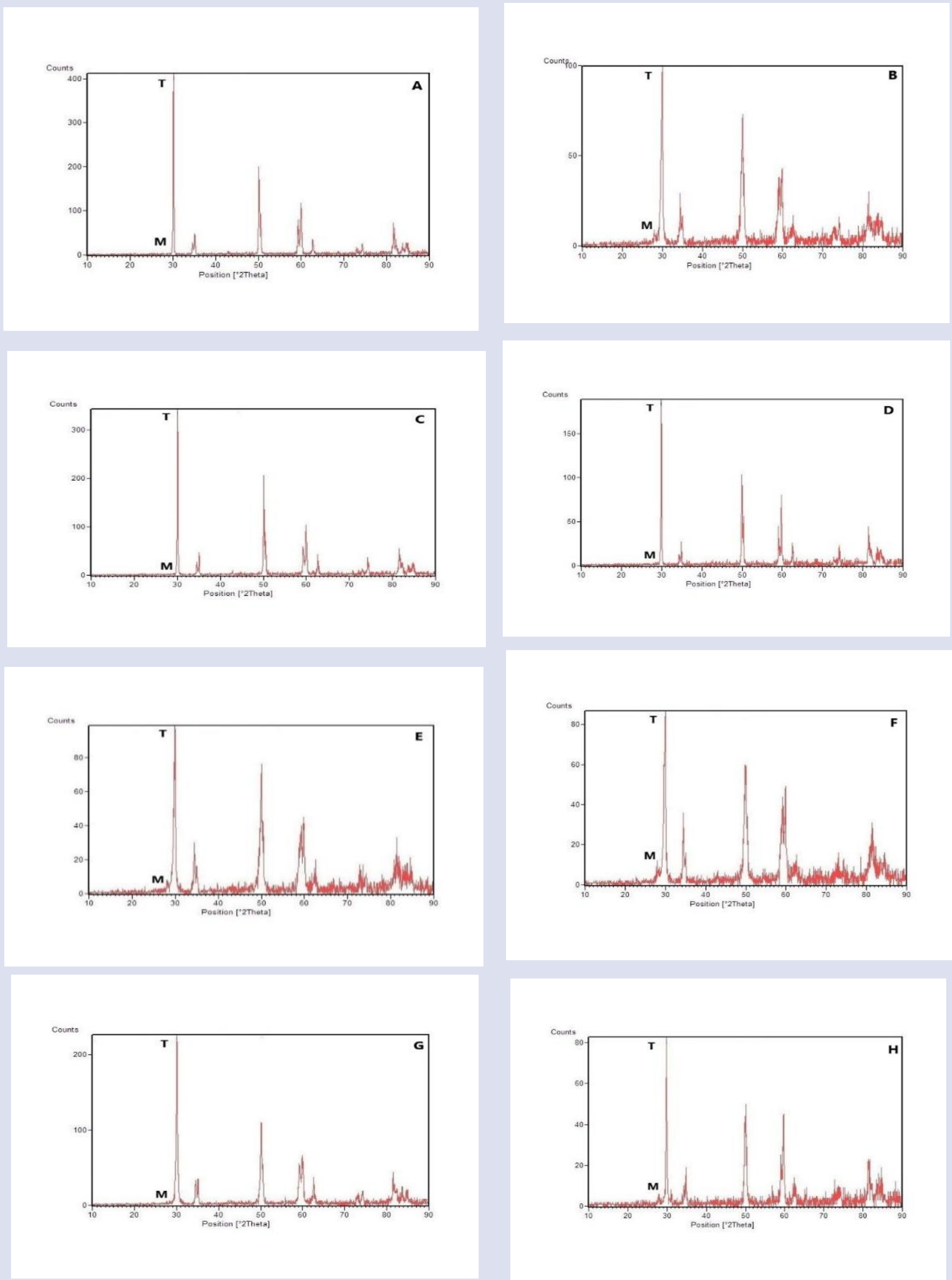

Figure 3. X-ray powder diffraction patterns of all sintered groups: A:Control, B:Sandblasting, C: Er:YAG Laser, D: Nd:YAG laser, E: Er:YAG Laser and Sandblasting, F: Nd:YAG Laser and Sandblasting, G: Fine Grained Bur, H: Coarse Grained Bur.