Original Article / Özgün Araştırma

Meandros Med Dent J
doi:10.4274/meandros.galenos.2023.59219
Meandros Med Dent J 2023;24(4):315-320
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Flexural Strength of Monolithic Zirconia After Zirconia-specific Grinding Procedures and Hydrothermal Aging

Zirkonyaya Özgü Aşındırma Prosedürleri ve Hidrotermal Yaşlandırma Sonrası Monolitik Zirkonyanın Eğilme Dayanımı

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Abstract

Objective: To evaluate the effect of grinding with different diamond burs on the surface roughness (Ra) and flexural strength (FS) of hydrothermally aged zirconia.

Materials and Methods: Ninety-eight bar-shaped monolithic zirconia specimens were prepared and divided into 7 subgroups according to grinding procedures: control, grinding with diamond burs (F; fine, M; medium, C; coarse); and grinding with zirconia-specific diamond burs (ZF; fine, ZM; medium, ZC; coarse). All ground specimens were polished using a two-step zirconia polishing system. All specimens were subjected to autoclave aging. Ra was measured using a profilometer. One specimen per group was examined by scanning electron microscopy and X-ray diffractometry. A 3-point FS test was performed using a universal testing machine.

Results: The lowest and highest Ra values were obtained in the control and C groups, respectively. The ZC group showed higher Ra values than the ZF and ZM groups. There was no difference between the FS values of the ZF and control groups. However, other grinding procedures led to decreased FS.

Conclusion: Zirconia-specific fine diamond burs are recommended to maintain the mechanical strength of zirconia when clinical adjustments are needed.

Keywords: Flexural strength, grinding, monolithic zirconia, surface roughness, zirconia-specific diamond bur

Öz

Amaç: Farklı elmas frezler ile yapılan aşındırmanın hidrotermal olarak yaşlandırılmış zirkonyanın yüzey pürüzlülüğü (Ra) ve eğilme dayanımına (FS) etkisini değerlendirmektir.

Gereç ve Yöntemler: Doksan sekiz adet bar şeklinde monolitik zirkonya örnek hazırlandı ve aşındırma prosedürlerine göre 7 gruba ayrıldı: kontrol, elmas frezlerle aşındırma (F; ince, M; orta, C; kalın), zirkonyaya özgü elmas frezlerle aşındırma (ZF; ince, ZM; orta, ZC; kalın). Aşındırma yapılan tüm örneklere 2 aşamalı zirkonya polisaj sistemi kullanılarak polisaj işlemi uygulandı. Tüm örnekler otoklavda yaşlandırıldı. Ra değerleri profilometre kullanılarak ölçüldü. Her gruptan bir örnek taramalı elektron mikroskobu ve X-ışını difraktometresi kullanılarak incelendi. Evrensel bir test cihazı kullanılarak 3 nokta eğme testi yapıldı.

Bulgular: En düşük ve en yüksek Ra değerleri sırasıyla kontrol ve C gruplarında elde edildi. ZC grubu ZF ve ZM gruplarından daha yüksek Ra değerleri gösterdi. ZF ve kontrol gruplarının FS değerleri arasında farklılık gözlenmedi. Ancak diğer aşındırma prosedürleri daha düşük FS değerlerine yol açtı.

Sonuç: Klinik düzenlemeler gerekli olduğunda zirkonyaya özel ince grenli elmas frezlerin kullanımı zirkonyanın mekanik dayanıklılığını korumak için önerilmektedir.

Anahtar Kelimeler: Eğilme dayanımı, aşındırma, monolitik zirkonya, yüzey pürüzlülüğü, zirkonyaya özgü elmas frez

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Received/Geliş Tarihi: 18.07.2023
Accepted/Kabul Tarihi: 17.10.2023

Introduction

Zirconium oxide has gained considerable popularity for prosthetic restorations due to its esthetic potential, mechanical properties, and biocompatibility. It is well-documented that one common clinical failure for zirconia-based restorations is the chipping of veneering porcelain (1,2). Monolithic zirconia is widely used to overcome this complication. These materials not only eliminate the need for veneering porcelain owing to their improved optical properties but also offer high flexural strength (FS), preservation of tooth structure during preparation, and reduced clinical and laboratory time (3).

Polycrystalline zirconia has three crystallographic phases: monoclinic (*m*), tetragonal (*t*), and cubic (*c*). It exists in *m*-phase at room temperature and *t*-phase between 1,170 ˚C and 2,370 ˚C. To retain the *t*-phase at room temperature, metal oxides, such as MgO, CaO, or Y_2O_3, are added to zirconium oxide, with yttria (Y_2O_3)-stabilized tetragonal zirconia polycrystal being the most commonly used type (4). However, surface treatments such as airborne particle abrasion and clinical adjustments using burs can trigger *t*→*m* phase transformation (5). This transformation also occurs when zirconia is exposed to the moist environment of the oral cavity. This phenomenon is referred to as low-temperature degradation, which may adversely affect the mechanical properties of zirconia ceramic (6-8).

Intraoral adjustments may be necessary for monolithic zirconia restorations (9,10). However, such adjustments with discs or burs can cause surface damage (5). Besides, chairside adjustments can increase surface roughness (Ra) on the monolithic zirconia restoration, resulting in undesirable conditions such as plaque accumulation and wear on opposing teeth (9,10). Intraoral polishing systems offer advantages over re-glazing, including reduced office visits and avoidance of multiple firing cycles. Moreover, zirconia polishing systems can reduce surface flaws and enhance the FS of restorations, thereby contributing to their longevity (10).

The influence of grinding and polishing procedures on the FS of zirconia is widely investigated in the literature (4,11). However, the effects of grinding with zirconia-specific diamond burs followed by manual polishing on the FS of monolithic zirconia are still unclear. Therefore, this study investigated the impact of grinding with either zirconia-specific or conventional diamond burs of varying grain sizes and polishing with zirconia-specific polishing systems on monolithic zirconia. The null hypotheses were that Ra and FS would remain unaffected by applying different bur types.

Materials and Methods

Specimen Preparation

The sample size was determined using a power analysis conducted with G*power software (v.3.1.9.2, Dusseldorf, Germany). With an effect size of 0.4, a significance level of 0.05, and a power of 80%, 14 specimens per group were determined sufficient. Ninety-eight bar-shaped specimens were obtained from a monolithic zirconia blank (CopraSmile, Whitepeaks Dental Solutions, Essen, Germany) using a diamond saw (Metcon 19-150, Metkon Instruments, Bursa, Turkey) mounted to a cutting device (MOD Dental, Esetron Smart Robotechnologies, Ankara, Turkey) under running water. The specimens were finished using 600, 1,000, and 1,200 grit silicon-carbide abrasive papers. The long edges of the bar-shaped specimens were chamfered using the final abrasive paper. All samples underwent ultrasonic cleaning in distilled water for 10 min. Specimens were sintered at 1,500 ˚C (Programat S1, Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions. The thickness of the samples for the grinding groups was considered 1.25 mm, according to the material to be removed during the process; sample thickness in the control group was adjusted to 1.2 mm. The width and length of sintered specimens were 4 mm and 20 mm, respectively. The specimens were randomly divided into 7 subgroups;

Control: No grinding and polishing

F: Grinding with fine diamond bur + polishing

M: Grinding with medium diamond bur + polishing

C: Grinding with coarse diamond bur + polishing

ZF: Grinding with zirconia-specific fine diamond bur + polishing

ZM: Grinding with zirconia-specific medium diamond bur + polishing

ZC: Grinding with zirconia-specific coarse diamond bur + polishing

Ethics committee approval was not obtained since the study was carried out in an experimental environment on materials that did not belong to any living organism.

Grinding and Polishing Procedures

Grinding was done using diamond burs and zirconia-specific diamond burs (Meisinger, Hager & Meisinger, Neuss, Germany) with a sweeping motion, removing 0.05 mm material from one entire surface of the specimen. A digital caliper was used to verify the final thickness of the specimens. Subsequently, manual polishing was performed on ground surfaces with a 2-step zirconia polishing system (Drendel + Zweiling Diamant, Kalletal, Germany) using each tool for 20 s. Grinding and polishing procedures were performed under water cooling. All procedures were conducted by a single experienced operator (H.Ş.). After polishing, all specimens were ultrasonically cleaned again in distilled water for 10 min. The burs used for grinding and the 2-step polishing system are shown in Figure 1.

Hydrothermal Aging

All specimens were subjected to an accelerated aging procedure using a steam autoclave (Yeson YS-22L-E, Ningbo Haishu Yeson Medical Device, Zhejiang, China).

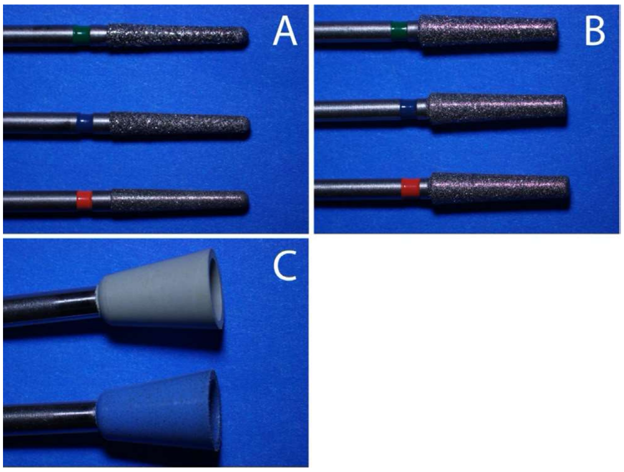

Figure 1. Grinding burs and polishing system A) Diamond grinding burs; B) Zirconia-specific diamond grinding burs; C) Zirconia-specific polishing system

The specimens were positioned in autoclave-safe trays and exposed to 5 sequential cycles, each lasting 60 min. Thus, the total exposure time amounted to 5 hours, maintaining a temperature of 134 °C and a pressure of 2 bars. Five-hour autoclave aging corresponds to approximately 15-20 years of actual aging (6,12).

Surface Roughness Evaluation

The average Ra values were obtained using a profilometer (Surftest SJ-210, Mitutoyo, Kanagawa, Japan). Measurements were taken at a constant speed of 0.5 mm/s and a cut-off value of λc=0.25 mm. The arithmetic mean of 5 perpendicular readings was accepted as the final Ra score for each specimen. The surface profilometer was recalibrated after measuring every 5 specimens.

Scanning Electron Microscopy

Additional samples from each group were analyzed using scanning electron microscopy (SEM). The samples were subjected to gold sputter-coating (Quorum Q150R ES, Quorum Technologies, East Grinstead, UK). Subsequently, SEM images were captured using the scanning electron microscope (EVO LS-10, Carl Zeiss Microscopy, Cambridge, UK) at magnifications of ×1000 and ×5000, operating at 25 kV.

X-ray Diffraction (XRD) Analysis

Crystal structure analysis was performed using an X-ray diffractometer (Bruker D8 Advance, Bruker AXS, Karlsruhe, Germany). Operating current and voltage conditions were set at 40 mA and 40 kV, respectively. Surface scans were performed within the range of 20 to 40 2θ degrees, using a step size of 0.019. The relative amount of *m*-phase (Xm) was determined by applying equation 1 (13), while the volumetric fraction (Fm) was calculated using equation 2 (14):

[1] $Xm=(Im(-111) + Im(111))/(Im(-111) + Im(111) + It(101))$

[2] $Fm=(1.311Xm)/(1+0.311Xm)$

where Im (−111) and Im (111) represent *m*-peak intensities at approximately 28 and 31 2θ°, respectively, and It (101) denotes *t*-peak intensity at approximately 30 2θ°.

Flexural Strength

FS data were obtained by implementing a three-point bending test using a universal testing machine (Marestek, Mares Engineering, İstanbul, Turkey). The specimens were positioned on metal supports with the treated surfaces under tension, and the force was applied to the center of samples with a constant cross-head speed of 1 mm/min until failure occurred. The radii of the 2 metal supports and loading piston were 0.8 mm, and the distance between the centers of the supports was 14 mm. FS values were calculated in MPa using the following equation based on ISO 6872:

$\sigma = 3Fd/2wh^2$

where σ is the FS; F is the fracture load (N); d is the span (distance between the center of the supports) (mm); w is the width of the specimen (mm); h is the thickness of the specimen (mm).

Statistical Analysis

Statistical analyses were performed at a significance level of α=0.05 (SPSS/PC Version 24.0; SPSS Inc., Chicago, IL, USA). The normality of the data was assessed using the Shapiro-Wilk test, while the homogeneity of variances was evaluated using the Levene test. To analyze both Ra and FS data, One-Way analysis of variance (ANOVA) and Tamhane's T2 tests were performed. Pearson correlation analysis was performed to determine the relationship between Ra and FS.

Results

Surface Roughness

One-way ANOVA revealed significant differences among subgroups (F=256.581, p<0.001). All treated groups showed significantly higher Ra values than the control group (p<0.001) (Table 1). For the diamond bur groups (F, M, and C), Ra increased statistically as the grain size increased. ZC showed statistically higher Ra values than both ZM and ZF (p<0.001). Although the mean Ra of ZM was higher than that of ZF, this difference was statistically insignificant (p=1,000). While there was no significant difference between F and ZF groups (p=0.448), medium and coarse zirconia-specific diamond burs led to decreased Ra compared to diamond burs with the same grain sizes (p<0.001).

SEM Analysis

In line with the Ra results, SEM images of the control group showed the smoothest surface (Figure 2). In contrast, the C group exhibited deep surface grooves and microcracks. The F group exhibited smoother surfaces than both C and M. The ZC group showed more irregular surfaces than both ZM and ZF.

Phase Transformation

No distinct *m*-peaks were observed in the control group (Figure 3), while ground specimens exhibited similar diffraction patterns with minimal *m*-peaks (Fm values= F: 2.9 %; M: 2.9%; C: 3.3%; ZF: 3%; ZM: 2.7%; ZC: 2.9%).

Flexural Strength

According to One-Way ANOVA, there were significant differences among test groups (F=191.126, p<0.001). All the ground groups, except ZF, showed significantly lower FS values than the control group (p<0.001) (Table 2). There was no significant difference between ZF and control groups (p=0.996). Zirconia-specific diamond bur groups showed statistically higher FS values than diamond bur groups with the same grain sizes (p<0.001). FS values decreased significantly with the increase in grain size for both diamond bur groups and zirconia-specific diamond bur groups (p<0.001).

Pearson correlation analysis revealed a significant negative correlation (r=-0.851, p<0.001) between Ra and FS.

Discussion

Based on the results obtained from this study, all null hypotheses were rejected due to significant differences among the test groups for the dependent variables.

Table 1. Results of the statistical analysis of surface roughness (Ra; µm)

	Mean ± SD*	Minimum	Maximum	95% CI
Control	0.384±0.047^{A}	0.29	0.46	0.357-0.411
ZF	0.697±0.051^{B}	0.58	0.78	0.667-0.726
ZM	0.716±0.051^{B}	0.66	0.85	0.687-0.746
F	0.768±0.099^{B}	0.59	0.89	0.710-0.825
ZC	0.934±0.049^{C}	0.87	1.02	0.906-0.962
M	0.982±0.079^{C}	0.85	1.14	0.936-1.028
C	1.254±0.050^{D}	1.12	1.32	1.225-1.283

SD: Standard deviation, CI: Confidence interval, *The groups with the same superscript letters are not statistically different (p>0.05)

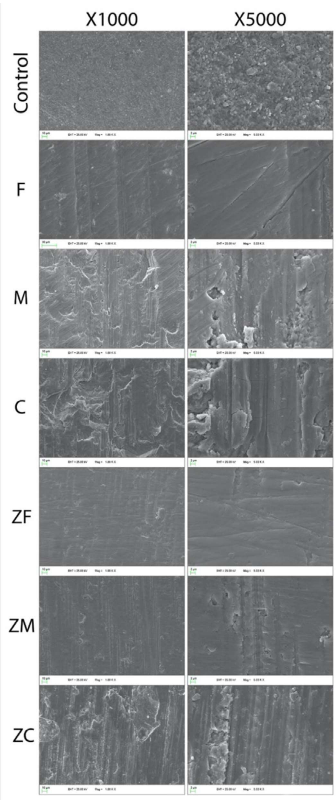

Figure 2. Scanning electron microscopy images

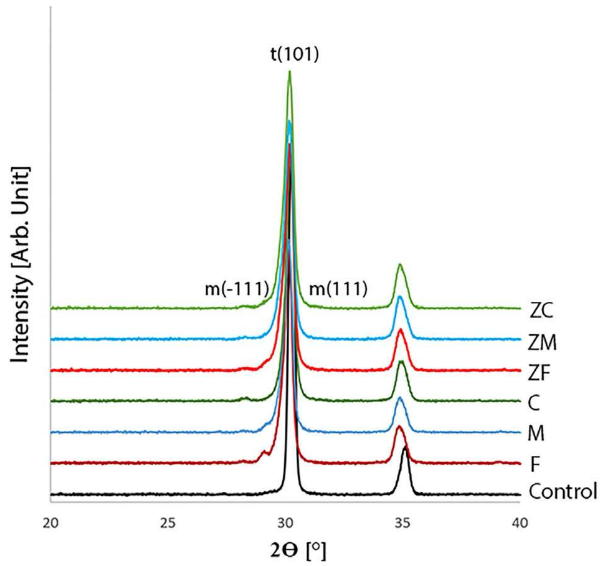

Figure 3. X-ray diffraction patterns

Table 2. Results of the statistical analysis of flexural strength (FS; MPa)

	Mean ± SD*	Minimum	Maximum	95% CI
C	310.19±11.32^{A}	294.71	329.47	303.65-316.73
M	357.96±21.10^{B}	327.24	386.17	345.78-370.15
ZC	372.20±22.20^{B}	327.21	420.49	359.39-385.02
F	401.48±22.95^{C}	358.77	445.02	388.23-414.73
ZM	440.58±32.34^{D}	387.91	493.36	421.91-459.26
ZF	533.57±34.90^{E}	473.75	590.41	513.42-553.72
Control	546.13±14.81^{E}	526.01	575.46	537.59-554.68

SD: Standard deviation, CI: Confidence interval, *The groups with the same superscript letters are not statistically different ($p>0.05$)

After chairside adjustments, the primary objective is to achieve a smooth surface comparable to the glazed surface, promoting oral tissue compatibility and resistance to plaque accumulation (15,16). Although glazed restorations display smooth surfaces, their wear behavior is not significantly superior to that of polished restorations. Glazed surfaces tend to result in more wear of the opposing teeth than polished surfaces (17,18). In a recent study, Badarneh et al. (19) preferred polishing over glazing as the surface finishing procedure for monolithic zirconia as it significantly reduced the wear of enamel antagonists.

In this study, Ra values were statistically higher in all grinding-applied groups than in the control group, even if the specimens were polished after grinding. Besides, Ra values significantly increased as the grain size increased in diamond bur and zirconia-specific diamond bur groups, except for the similarity between ZF and ZM groups. Corroborating this result, Hmaidouch et al. (1) concluded that coarse grinding is closely related to high roughness values. In line with the Ra results, SEM analyses of the current study revealed that coarse grinding caused more distinct grooves than grinding with medium and fine burs.

Various factors, such as phase change, crack formation, and surface flaws, can determine the mechanical strength of zirconia (20). In earlier research, Kosmac et al. (21) concluded that the relation between the depth of grinding-induced surface compressive layer resulting from phase transformation and the length of surface flaws was critical. According to the same authors, when the length of surface flaws exceeded the thickness of the surface compressive layer, the mechanical strength of zirconia tended to decrease. Microcracks or flaws due to surface grinding act as sites of stress concentration, which may cause a reduction in the FS of zirconia (22). In the present study, the mean FS of the grinding-applied groups, except ZF, was lower than the control group. However, no significant difference was observed between the FS of ZF and control groups, probably due to the low Ra values of ZF and few surface defects seen in SEM images. Therefore, grinding with zirconia-specific fine diamond burs followed by polishing may be a promising protocol if clinical adjustments are needed. Conversely, the C group showed the lowest mean FS, which may be strongly related to microcracks, as seen in SEM images. Moreover, FS values significantly decreased as the grain size increased in diamond bur and zirconia-specific diamond bur groups. Similarly, some studies highlighted that excessive grinding could lead to deep surface flaws (20,23). Therefore, clinicians should avoid coarse grinding of monolithic zirconia restorations.

To simulate long-term intraoral conditions, accelerated hydrothermal aging was applied to all zirconia specimens, which can be also effective on mechanical properties (24). However, XRD analysis showed no distinct *m*-peaks for the control group. This result indicated that hydrothermal aging did not trigger $t \rightarrow m$ phase transformation, possibly due to the high yttria content of the monolithic zirconia used. Moreover, each type of diamond bur led to similar XRD patterns. Fm values of ground specimens ranged between 2.7% and 3.3%. Thus, the FS of samples may not have been adversely affected by grinding-induced minimal $t \rightarrow m$ phase transformation.

In a recent study, Kheur et al. (16) used diamond and modified diamond burs (zirconia specified) for zirconia cutting. The results showed no relationship between mean Ra and FS. Lee et al. (4) reported that coarse grinding without subsequent polishing resulted in higher Ra and lower FS than fine grinding. The current study showed a meaningful negative correlation between Ra and FS. Moreover, grinding with diamond burs, except fine grinding, resulted in higher Ra values than grinding with zirconia-specific diamond burs with the same grain size; FS values were statistically lower in diamond bur groups compared to zirconia-specific bur groups.

In this study, chairside adjustment procedures were simulated by a single operator. Despite efforts to standardize the grinding and polishing procedures and maintain consistent pressure, the pressure applied was possibly not as precisely controlled as in a controlled experimental setup. Another limitation of the study was that crucial factors, such as dynamic occlusal load, neuromuscular forces, and parafunctional habits, were excluded. The present study focused on the FS of a single brand of zirconia that consists of approximately 9% yttria. Therefore, additional studies evaluating different types of zirconia are needed.

Conclusion

From a clinical perspective, chairside adjustments of monolithic zirconia restorations should be avoided. However, we recommend zirconia-specific diamond burs with smaller grain sizes when occlusal adjustments are necessary for achieving optimal occlusal harmony. This approach may ensure long-term durability without jeopardizing the mechanical properties of monolithic zirconia.

Ethics

Ethics Committee Approval: Ethics committee approval was not obtained since the study was carried out in an experimental environment on materials that did not belong to any living organism.

Informed Consent: Informed consent is not required.

Peer-review: Externally and internally peer-reviewed.

Authorship Contributions

Surgical and Medical Practices: H.Ş., Ş.K., Concept: Y.O., M.T.Y., Design: Y.O., M.T.Y., Data Collection or Processing: H.Ş., Y.O., Analysis or Interpretation: Y.O., Literature Search: H.Ş., Ş.K., Writing: H.Ş., Y.O.

Conflict of Interest: No conflict of interest was declared by the authors.

Financial Disclosure: The authors declared that this study received no financial support.

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