

Effects of Sintering Temperature and Finishing Procedure on Surface Roughness of Different Yttria Content Zirconia

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ABSTRACT

Introduction: Zirconia-based ceramics have been successfully used to fabricate dental prostheses using the dental Computer-Aided Design/Computer-Aided Manufacturing (CAD/CAM) system. Fabrication and finishing of new generation zirconia ceramics is an important issue for the success of restorations. It is necessary to investigate whether these processes affect the surface properties of zirconia materials.

Aim: To investigate the effects of different sintering temperatures and finishing procedures on the surface roughness of zirconia.

Materials and Methods: This in-vitro study was carried out in the Erciyes University Research Laboratory from 7th January to 4th April 2021. Katana Ultra Translucent Multi Layered (KT10; Kuraray Noritake Dental Inc., Tokyo, Japan) and Zolid High Translucent+Preshade (AmannGirrbach AG, Herrschaftswiesen, Austria) were the two types of zirconia materials used in this in-vitro study. Total 40 samples (20 in each group) were produced using the dry program in the InLAB MC X5 milling machine

(Dentsply Sirona, Bensheim, Germany). Total 20 specimens (10 from each group) were sintered in a furnace for 2 hour at 1450°C and the remaining 20 specimens (10 from each group) were sintered at 1650°C. The two surfaces of the specimens were divided into two groups; one surface of the specimens underwent polishing and the other underwent glazing. A profilometer was used to analyse the surface roughness of the specimens. Three-way analysis of variance was used to assess significant differences in the average roughness values based on the material, sintering temperature, finishing procedure.

Results: The surface roughness values differed between specimens that underwent the glazing and the polishing process at high sintering temperatures. The glazed samples (1.154 µm) had higher surface roughness values than the polished samples (0.808 µm) at 1650°C.

Conclusion: The polishing process is recommended instead of glazing as a finishing procedure for zirconia materials.

Keywords: Ceramics, Computer-aided manufacturing, Glazing, Profilometer

INTRODUCTION

Zirconia ceramics display good mechanical properties and high biocompatibility and are widely following the development of the Computer-Aided Design and Computer-Aided Manufacturing (CAD/CAM) technology. Additives such as yttria (3 mol%) were added to zirconia ceramics to stabilise the tetragonal crystal phase with the Yttria Tetragonal Zirconia Polycrystals (3Y-TZP) and to improve the aesthetics [1]. The translucent properties of first-generation 3Y-TZPs were subsequently improved by including a cubic phase, raising the yttria content, removing the porosity by sintering at high temperature, and reducing the concentration of Al₂O₃ (from 0.25% to 0.1%) [2]. On the other hand, the strength and toughness of the material were decreased due to the stress-induced transformation of the zirconia ceramic [3]. The mechanical properties of zirconia ceramics with 5-8 mol% yttria were affected due to the cubic phase and the increased yttria content [1,4]. The sintering temperature affects the mean grain size and pore diameter of zirconia [5], enhances the physical properties of the material [6], and markedly affects its roughness [7].

Finishing procedures are important for the long-term success of zirconia ceramics. These procedures not only improve the appearance of the restoration, but also provide compatibility with the surrounding tissues, reduce antagonistic teeth wear, and prevent bacterial invasion [8]. It is highly recommended to smoothen the rough surfaces on the zirconia ceramic restoration [9,10]. Rough surfaces can cause antagonistic tooth wear, plaque accumulation, patient discomfort; additionally, it can adversely affect the aesthetics when compared to polished surfaces [11]. Polishing results in a shiny and smooth surface. Glazing is a common procedure performed to achieve an aesthetic glass-covered look on the surface of the

restoration. Glazing helps to achieve a natural tooth-like shine. It provides surface smoothness by reducing the depth and width of the surface defect [12,13].

Generally, the same finishing procedures and sintering temperatures are applied to zirconia ceramics, despite variations in the yttria content [14,15]. Fabricated and final procedures should be examined according to the surface characteristics of zirconia materials. The aim of this study was to evaluate the surface properties of the zirconia ceramics and to examine the effect of the sintering temperature on the surface roughness after glazing and polishing. The null hypothesis was that the surface roughness would not be affected by the different finishing procedures and sintering temperatures.

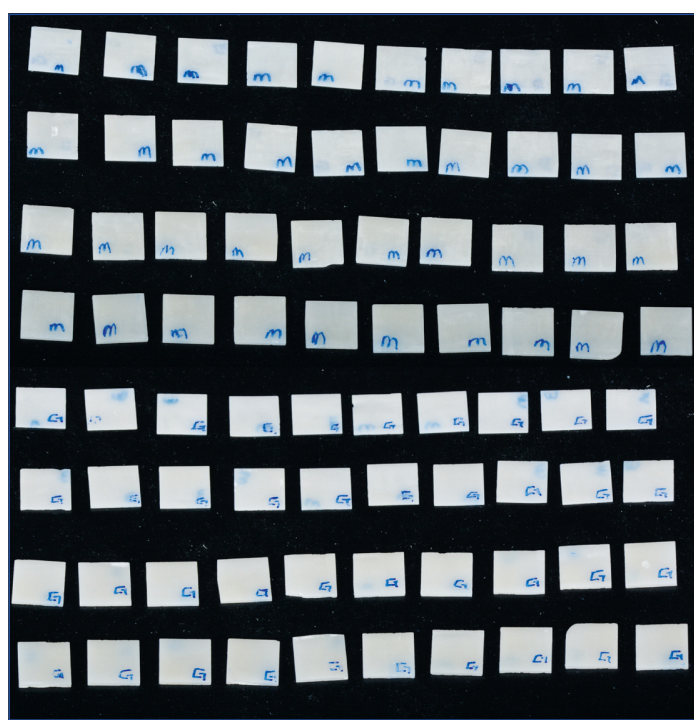
MATERIALS AND METHODS

This in-vitro study was carried out in the Erciyes University research Laboratory from 7th January to 4th April 2021. Two types of zirconia ceramics were used in this in-vitro study were Katana Ultra Translucent Multi Layered (UTML; KT10; Kuraray Noritake Dental Inc., Tokyo, Japan) containing 5 mol% yttria and Zolid High Translucent (HT)+ Preshade (AmannGirrbach AG, Herrschaftswiesen, Austria) containing 6-7 mol% yttria [Table/Fig-1]. Three-Dimensional (3D) images were created using the Computer-Aided Design (CAD) software program (dimensions, 12×12 mm square; thickness, 1 mm) [Table/Fig-2]. The image was saved as an STL (STereoLithography) file and sent to the milling unit.

A total of 40 samples (20 from the Katana UTML block and 20 from the Zolid HT+ preshade block) were produced using the InLAB MC X5 milling machine (dry program; Dentsply Sirona, Bensheim, Almany). The connection parts of the milled samples were corrected before sintering. The specimens were then sintered in a furnace

Material	Classification	Composition	Manufacturer	Sinter temperature	n
Katana Ultra Translucent Multi Layered (Katana UTML)	5 mol% yttria content zirconia	ZrO ₂ +HfO ₂ : 87-92% Y ₂ O ₃ : 8-11% Others: 0-2%	Kuraray Noritake Dental Inc., Tokyo, Japan	At 1450°C with a duration of 2 hours for heating.	10
Katana Ultra Translucent Multi Layered (Katana UTML)	5 mol% yttria content zirconia	ZrO ₂ +HfO ₂ : 87-92% Y ₂ O ₃ : 8-11% Others: 0-2%	Kuraray Noritake Dental Inc., Tokyo, Japan	At 1650°C with a duration of 2 hours for heating.	10
Zolid High Translucent+ Preshade (Zolid HT+ Preshade)	6-7 mol% yttria content zirconia	ZrO ₂ + HfO ₂ + Y ₂ O ₃ : ≥ 99.0 Y ₂ O ₃ : 6,0 - 7,0 HfO ₂ : ≤5 Al ₂ O ₃ : ≤0.5 Other oxides: ≤1	AmannGirrbach AG, Herrschaftswiesen, Austria	At 1450°C with a duration of 2 hours for heating.	10
Zolid High Translucent+ Preshade (Zolid HT+ Preshade)	6-7 mol% yttria content zirconia	ZrO ₂ + HfO ₂ + Y ₂ O ₃ : ≥ 99.0 Y ₂ O ₃ : 6,0 - 7,0 HfO ₂ : ≤5 Al ₂ O ₃ : ≤0.5 Other oxides: ≤1	AmannGirrbach AG, Herrschaftswiesen, Austria	At 1650°C with a duration of 2 hours for heating.	10
EVE polishing kit	Polishing equipment	Synthetically bonded grinder interspersed with diamond	EVE Diacera, EVA Ernst Vetter GmbH, Pforzheim, Germany	At 1450°C and 1650°C with a duration of 2 hours for heating.	40
Ivoclar glaze powder	Glaze powder	SiO ₂ ;CaO;AlO ₃ ;CeO ₂ ; Na ₂ O; K ₂ O;B ₂ O ₃ ;ZnO;F;Li ₂ O;ZrO ₂ ; SrO; TiO ₂	Ivoclar Vivadent AG, Liechtenstein	At 1450°C and 1650°C with a duration of 2 hours for heating.	40
Ivoclar glaze liquid	Glaze liquid	Pentandiol	Ivoclar Vivadent AG, Liechtenstein	At 1450°C and 1650°C with a duration of 2 hours for heating.	40

[Table/Fig-1]: Materials used in the present study.



[Table/Fig-2]: The samples prepared for the present study.

(In Therm, Amann Girrbach AG, Germany) at 1450°C (n=10 from each group; Temperature A) and 1650°C (n=10 from each group; Temperature B) with a duration of 2 hours for heating.

Subsequently, one surface of the specimens underwent polishing and the other underwent glazing. The mechanical polishing was performed using the EVE polishing kit (EVE Diacera, EVA Ernst Vetter GmbH, Pforzheim, Germany) and a universal polishing paste (Renfert Polish, Renfert GmbH, Germany) at a speed of 10,000 rpm for 30 seconds, as described previously [16-18]. The polishing was accomplished by a single trained operator (CD) using a handpiece (low speed) at an angle of 90°. The other surface was glazed (Ivoclar Glaze, Ivoclar Vivadent AG, Liechtenstein) and fired in a porcelain furnace (Programat P310, Ivoclar Vivadent AG, Liechtenstein) using the glazing program, according to the manufacturer's instructions. Both the surfaces of the specimens were cleaned using a steam device (Gazella, Gold dental, Silter, Turkey). A profilometer (SurfTest SJ-310 Mitutoyo, ABD) was used to analyse the surfaces of the zirconia specimens. Three measurements were obtained from each of the two surfaces from each specimen and the average surface roughness values (Ra) were calculated.

STATISTICAL ANALYSIS

Three-way analysis of variance was used to assess significant differences in the average roughness values based on the material, sintering temperature, finishing procedure. The Duncan test was used to compare each groups. The data were tested for normal distribution using the Shapiro-Wilk test. The statistical analysis was performed using IBM Statistical Package for the Social Sciences (SPSS) version 23.0, and the significance level was set as p-value <0.05.

RESULTS

The average surface roughness of all the groups is given in [Table/Fig-3]. The Zolid HT+ preshade zirconia presented with less surface roughness than the Katana UTML zirconia at low sintering temperatures (temperature A). The average surface roughness value of the Zolid HT+ preshade zirconia was lower than that of the Katana UTML zirconia in the group that underwent the glazing procedure at high sintering temperatures (temperature B). Alternatively, the Katana UTML zirconia specimens showed lower average surface roughness values than the Zolid HT+ preshade zirconia specimens in the group that underwent polishing at high sintering temperatures (temperature B). The least roughness value (0.795 µm) was observed in the Katana UTML zirconia material that was polished at temperature B. The highest roughness value (1.205 µm) was observed

Sintering temperature	Finishing procedure	Materials (Ra)		Total Roughness averages (Ra)
		Zolid-High translucent + Preshade	Katana ultra translucent multi layered	
Temperature A (1450°C)	Glazing	0.848±0.271	1.143±0.355	0.996±0.343 ^{ab}
	Polishing	0.929±0.210	1.023±0.287	0.976±0.250 ^{ab}
	Total	0.889±0.240	1.083±0.320	0.986±0.296
Temperature B (1650°C)	Glazing	1.102±0.398	1.205±0.347	1.154±0.367 ^a
	Polishing	0.821±0.404	0.795±0.238	0.808±0.323 ^a
	Total	0.962±0.416	1.000±0.358	0.981±0.384
Total	Glazing	0.975±0.356	1.174±0.343	1.075±0.360
	Polishing	0.875±0.318	0.909±0.282	0.892±0.297
	Total	0.925±0.337	1.042±0.338	0.983±0.341

[Table/Fig-3]: Statistical data on surface roughness values according to material, sinter temperature and finishing procedure. (a-b: There is no difference between the same letter interactions). {Duncan test result is indicated by letters. Values in the glazed specimens at temperature B were higher than those polished specimens at the same temperature (p=0.013)}

in the Katana UTML zirconia material that was glazed at temperature B. significant association was found between the finishing procedures and roughness values ((p-value=0.013) [Table/Fig-4]. The average surface roughness value for all groups after glazing was 1.075 μm , and that after polishing was 0.892 μm . The association between the sintering temperature and the surface treatments was found to be statistically significant on the roughness values (p-value=0.026). At temperature A, the average value of the glazed surface was 0.996, while that of the polished surface was 0.976 μm ; likewise, at temperature B, the average value of the glazed surface was 1.154 μm , and that of the polished surface was 0.808 μm . The main effects of the material and the sintering temperature did not have any statistically significant effect on the roughness values (p-value=0.281). Likewise, interactions among the material, sintering temperature, and surface treatments did not significantly affect the roughness values.

Variables of interest	Degrees of freedom (df)	Sum of squares	Mean square	f-value	p-value
Materials	1	0.271	0.271	2.632	0.109
Sintering temperature	1	0.001	0.001	0.005	0.945
Finishing procedures	1	0.666	0.666	6.459	0.013*
Materials and sintering temperature	1	0.122	0.122	1.180	0.281
Materials and finishing procedures	1	0.136	0.136	1.320	0.254
Sintering temperature and finishing procedures	1	0.531	0.531	5.152	0.026*
Materials and sintering temperature and finishing procedures	1	0.006	0.006	0.063	0.803

[Table/Fig-4]: Comparison of roughness values in terms of group, sinter temperature and finishing procedure.

Three-way analysis of variance test; (*p-value <0.05 was considered as significant)

DISCUSSION

This study assessed the effects of glazing and polishing and the sintering temperature on the surface roughness of zirconia ceramics. Significant differences in the roughness values were observed between specimens that underwent the two different finishing procedures, thus rejecting the null hypothesis of this study.

Differences in the yttrium content could induce different surface characteristics, such as variations in the grain size and the phase proportions [3, 19-21]. An increase in the grain size has been reported

when the sintering temperature is increased [22,23]. Amarante JEV et al., reported an inversely proportional relationship between the tetragonal and cubic grain size and the surface roughness [24]. In the current study, no significant difference was found in terms of the surface roughness between the two zirconia materials with different yttria content. However, differences in surface roughness have been reported in other studies [2,8,14]. This discrepancy in the results could be attributed to the use of different polishing protocols and systems. One study reported similar results in specimens that underwent polishing and glazing [2]. In the present study, no significant difference between the polishing (0.976 μm) and glazing (0.996 μm) processes at sintering temperature A was observed. Some zirconia polishing systems are known to produce surfaces that are considerably smoother than other systems [18,25]. In the present study, the surface roughness values of the polishing surfaces were lower than those of the glazed surfaces. Polishing instead of glazing has been reported to reduce the surface roughness of monolithic zirconia [26-28]. A glazed surface is reported to break during wear because it is weaker and softer than the zirconia surface [29]. In cases where an aesthetic glazed restoration is required, it is recommended to polish the surface before glazing [2,10]. Moreover, the surface roughness of a damaged glazed layer was reported to be higher than that of a non glazed layer [30]. In the present study, the glazed samples had higher surface roughness values than the polished samples at 1650°C.

No significant differences in the surface roughness values of zirconia ceramic samples have been reported based on variations in the sintering parameters [31-33]. Similarly, the different sintering parameters did not appear to affect the surface roughness in the current study. Inokoshi M et al., reported that variations in the sintering temperature and holding time increased the cubic phase in the surface of the structure; the cubic phase was increased due to the increase in the two parameters [34]. Preis V et al., reported that the cubic phase increases when the sintering temperature is increased above 1450°C [29]. Some studies have reported the presence of large cubic grains in zirconia at high sintering temperatures [34-37]. In the current study, the interaction between the sintering temperature and the finishing procedure used was found to have a statistically significant effect on the roughness value. However, this might be related to the difference between the polished and the glazed surfaces. No significant association was observed between the material content and the surface roughness. Comparison between various studies has been done in [Table/Fig-5] [2,8,14,25,32,33].

Author's name and year of publication	Place of study	Sample size	Materials considered	Parameters assessed	Conclusion
Khayat W et al., 2018 [2]	Tufts University School of Dental Medicine, Boston, Mass	15x1.5 mm zirconia disks	High translucency zirconia (Tizian Blank Translucent 98-mm Zirconium; Schütz)	Surface roughness values after grinding (1,70 μm), after polishing with Brasseler (1,0 μm), after polishing with Komet (0.81 μm) and after glazing (0.79 μm).	Roughness averages values of the groups after grinding and polishing were significantly different between the other groups.
Goo C et al., 2016 [8]	University Kebangsaan, Kuala Lumpur, Malaysia	7 mm longx7 mm widex2 mm thick zirconia specimens	High Translucency Zirconia (LAVA PLUS, 3M ESPE, St Paul, MN, USA)	Mean Ra values after treatment with the various polishing systems ranged from 0.24 to 0.51 μm .	The use of zirconia polishers is thus recommended for polishing zirconia prostheses after clinical and laboratory adjustments.
Shin HS and Lee JS, 2021 [14]	Dankook University, Dankook Dental Hospital, Dongnamgu, Cheonan 31116, Republic of Korea	Bar-shaped specimens were fabricated as 12.0x6.0x4.0 mm	IPS e.max ZirCAD LT, IPS e.max ZirCAD MT, IPS e.max ZirCAD MT multi (Ivoclar Vivadent AG, Schaan, Liechtenstein)	IPS e.max ZirCAD MT showed the highest surface roughness (0.19±0.05 μm).	Zirconia with different yttrium oxide compositions showed similar surface roughness after polished with zirconia polishing burs.
Hatanaka GR et al., 2020 [25]	São Paulo State University (UNESP), Araraquara, Brazil	25x5.0x1.5 mm bar shape (before sintering) 20x4.0x1.2 mm bar shape (after sintering)	Partially stabilised zirconia (Prettau; Zirkozahn), Fully stabilised zirconia (Prettau Anterior; Zir-konzahn)	Surface roughness of Partially stabilised zirconia: Grinding and glaze (0,97 μm). Grinding and polishing (2.12 μm). Surface roughness of Fully stabilised zirconia: Grinding and glaze (1,21 μm). Grinding and polishing (2.29 μm).	Grinding increased the roughness of both ceramics. Polishing after grinding led to a smoother surface without compromising the flexural strength.

Preis V et al., 2014 [32]	Regensburg University Medical Centre, Regensburg, Germany	Diameter 20 mm, thickness 1.5 mm discs	Two different yttria-stabilised zirconia ceramics Cercon high translucency was sintered at 1500°C (DeguDent, Hanau, G). Cercon base was sintered at 1350°C (DeguDent, Hanau, G)	When comparing the zirconia materials Cercon base and Cercon ht, specimens with the same surface treatment did not show any significant (p-value >0.05) differences in roughness, neither before nor after wear testing.	Careful polishing including all polishing steps reduced the surface roughness of ground zirconia. In clinical application, zirconia should be polished according to the instructions of the manufacturer to keep values for surface roughness and phase transformation as low as possible.
Öztürk C and Can G, 2019 [33]	Hatay Mustafa Kemal University, Hatay, Turkey	The final dimension of the samples was 15.5×12.5×1.2±0.03 mm	Translucent monolithic zirconia materials. Group TZI (Incoris TZI C, Sirona Dental Systems GmbH, Bensheim, Germany) Group Up (Upcera, Shenzhen Upcera Co., Ltd, Shenzhen, China) were used.	No significant differences were found between the control group and subgroups in surface roughness values (p-value >0.05).	It was concluded that sintering parameters did not significantly affect the microstructure of translucent monolithic zirconia. Changes in the sintering parameters did not significantly affect the surface roughness of the translucent monolithic zirconia. For all the groups, the surface roughness was above the clinically acceptable values. Changes in sintering parameters did not significantly affect the flexural strength of the translucent monolithic zirconia.
Present study (2022)	Istanbul Medipol University, Istanbul, Turkey	Dimensions of samples; 12×12 mm square; thickness, 1 mm	Katana Ultra Translucent Multi Layered (UTML; KT10; Kuraray Noritake Dental Inc., Tokyo, Japan) containing 5 mol% yttria and Zolid HT+ Preshade (AmannGirrbach AG, Herrschaftswiesen, Austria) containing 6-7 mol% yttria. The connection parts of the milled samples were corrected before sintering. The specimens were then sintered in a furnace (In Therm, Amann Girrbach AG, Germany) at 1450°C (n=10 from each group; Temperature A) and 1650°C (n=10 from each group; Temperature B) with a duration of 2 hours for heating.	The surface roughness values differed between specimens that underwent the glazing and the polishing process at high sintering temperatures (1650°C). The glazed samples (1,154 µm) had higher surface roughness values than the polished samples (0,808 µm) at 1650°C.	Differences in yttria content and different sintering temperatures did not affect the surface roughness of the zirconia ceramics. Polishing of the surface of the zirconia ceramic significantly reduced the roughness value when compared to glazing. These findings indicate that polishing is recommended as a finishing procedure for zirconia materials. The finishing procedures of new generation zirconia materials should be examined using more groups and the effect of the same material by the differences in the production process should be evaluated.

[Table/Fig-5]: Description and summary of included literature data [2,8,14,25,32,33].

Limitation(s)

One of the limitations of this in-vitro study is the small sample size and the low number of parameters tested. The polishing process can be affected by several other factors such as pressure, rotations per minute, and time.

CONCLUSION(S)

Within the limitations of this in-vitro study, differences in yttria content and different sintering temperatures did not affect the surface roughness of the zirconia ceramics. Polishing of the surface of the zirconia ceramic significantly reduced the roughness value when compared to glazing. These findings indicate that polishing is recommended as a finishing procedure for zirconia materials.

The finishing procedures of new generation zirconia materials should be examined using more groups and the effect of the same material by the differences in the production process should be evaluated. Additional studies are required to determine whether variations in sintering temperature can influence the surface of the material based on its content.

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