

Article

Not peer-reviewed version

The Effect of Different Sintering Protocols on the Mechanical and Microstructural Properties of Two Multilayered Zirconia Ceramics: An In Vitro Study

[Lana Alatrash](#)^{*} and Asude Dilek Nalbant

Posted Date: 24 October 2025

doi: 10.20944/preprints202510.1872.v1

Keywords: sintering; grain size; mechanical properties; ZrO₂



Preprints.org is a free multidisciplinary platform providing preprint service that is dedicated to making early versions of research outputs permanently available and citable. Preprints posted at Preprints.org appear in Web of Science, Crossref, Google Scholar, Scilit, Europe PMC.

Copyright: This open access article is published under a Creative Commons CC BY 4.0 license, which permit the free download, distribution, and reuse, provided that the author and preprint are cited in any reuse.

Disclaimer/Publisher's Note: The statements, opinions, and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions, or products referred to in the content.

Article

The Effect of Different Sintering Protocols on the Mechanical and Microstructural Properties of Two Multilayered Zirconia Ceramics: An In Vitro Study

Lana Alatrash ^{1,*} and Asude Dilek Nalbant ²

¹ Research Assistant, Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara, Turkey

² Professor, Department of Prosthodontics, Faculty of Dentistry, Gazi University, Ankara, Turkey

* Correspondence: lanaatrash17@gmail.com

Abstract

This study evaluated the effects of different sintering protocols on the mechanical and microstructural properties of two multilayered zirconia materials: strength-gradient zirconia (KATANA YML) and color-gradient zirconia (KATANA UTML). Bar-shaped specimens were fabricated from both zirconia types. Three sintering protocols were applied: manufacturer recommended conventional (7 h at 1550 °C), high-speed (54 min at 1600 °C), and a modified high-speed protocol (51 min at 1600 °C). Eighty-four specimens underwent three-point flexural strength testing. SEM and XRD analyses were used to assess microstructure and phase composition. No significant differences in flexural strength were found among sintering protocols ($p > 0.05$), but YML consistently showed higher strength than UTML ($p < 0.05$). The highest strength in YML was observed after high-speed sintering, followed by the shortened and conventional protocols. In UTML, the modified protocol yielded the highest strength, followed by the high-speed and then conventional protocol. SEM revealed finer, more homogeneous grains with shorter sintering times. XRD confirmed stable phase composition across all protocols. High-speed and modified high-speed sintering protocols can reduce processing time without compromising zirconia's mechanical performance. Material type had a greater effect on flexural strength than sintering time, though microstructure was protocol dependent. Proper selection of zirconia type and sintering strategy is essential for optimal outcomes.

Keywords: sintering; grain size; mechanical properties; ZrO_2

1. Introduction

The advancement of computer-aided design (CAD) and computer-aided manufacturing (CAM) systems, along with their user-friendly applications, has led to innovative strategies in modern prosthodontic treatments[1]. The integration of high-performance materials, such as zirconia ceramics, with emerging technologies has significantly reshaped both dental education and clinical practice [2].Yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) is widely recognized for its durability, biocompatibility, and corrosion resistance. Zirconia exists in three crystallographic phases depending on temperature: monoclinic at room temperature, tetragonal above ~1170 °C, and cubic above ~2370 °C. The tetragonal phase provides the highest mechanical strength and fracture toughness [3]. To retain this phase at room temperature, zirconia is doped with stabilizing oxides such as yttrium (Y), cerium (Ce), calcium (Ca), or magnesium (Mg) [4]. Among these, yttria is particularly effective in maintaining phase stability, limiting grain growth, and enhancing thermal resistance [5].

Yttria-stabilized zirconia is classified based on the molar concentration of yttria—3Y-TZP, 4Y-TZP, 5Y-TZP, and 6Y-TZP—where higher yttria content increases translucency by promoting the cubic phase, though at the expense of mechanical strength due to reduced transformation toughening

[6–8]. Recently, multilayer zirconia systems with gradient yttria content (e.g., 3Y-5Y or 4Y-5Y) have been introduced to balance strength and esthetics within a single restoration. These systems typically feature lower yttria concentrations (enhancing strength) in cervical and middle zones, and higher yttria (for translucency) in the occlusal zone. However, the inclusion of cubic-rich, lower-strength zirconia in load-bearing areas may lead to crack propagation and chipping under occlusal forces [6,9].

Sintering is a key factor influencing the phase stability, microstructure, and mechanical behavior of zirconia [10–12]. With recent technological advancements, novel speed sintering protocols have been developed to dramatically shorten processing times, sometimes to just a few minutes, compared to conventional sintering [13]. These protocols primarily manipulate temperature and duration, both of which affect mechanical, optical, and microstructural characteristics of zirconia ceramics [10,14,15]. Speed sintering can effectively reduce grain growth and maintain material density, thereby preserving desirable mechanical and structural properties [12,16,17]. Therefore, the aim of this in vitro study was to evaluate the impact of three sintering protocols conventional, manufacturer-recommended high speed, and experimental speed on the flexural strength, microstructure, and phase composition of two multilayer monolithic zirconia materials. The null hypotheses were shortening the sintering time would not significantly affect the mechanical and microstructural properties of the materials.

2. Materials and Methods

2.1. Materials Used

Two commercially available pre-shaded multilayer monolithic zirconia discs were selected for this study. One was a strength-gradient zirconia with varying yttria (Y₂O₃) concentrations across layers (KATANA™ YML, 18 mm, NW), and the other was a color-gradient zirconia with a uniform yttria distribution throughout (KATANA™ UTML, 18 mm, ENW), both provided by Kuraray Noritake Dental Inc. (Japan). The compositional differences between the two materials are presented in Table 1.

Table 1. Composition and Characteristics of the Zirconia Materials Used in This Study.

Material	Trade Name	LOT No	Manufacturer	Y ₂ O ₃ content (nominal)
Layered Monolithic Zirconia	KATANA™ YML (18 mm, NW)	EITAY	Kuraray Noritake Dental Inc., Japan	Multilayer: dentin ≈ 3Y; transition ≈ 4Y; enamel ≈ 5Y
Layered Monolithic Zirconia	KATANA™ UTML (18 mm, ENW)	ENCJF	Kuraray Noritake Dental Inc., Japan	Uniform: ≈ 5Y

Note: The manufacturer does not provide exact Y₂O₃ ratios for YML and UTML. For YML, the nominal compositions are reported in the literature: dentin ≈ 3Y, transition ≈ 4Y, and enamel ≈ 5Y (Strasser et al., [18]). Because YML is multilayered, a single bulk value cannot represent the entire structure. For UTML, classification studies indicate ≈ 5 mol% Y₂O₃ (≈ 13 wt%), which is also in line with our EDS results (Kongkiatkamon et al.,[6]).

2.2. Specimen Preparation

A total of 96 rectangular zirconia specimens were prepared for this study. The final dimensions were 4.0 ± 0.2 mm (width), 15.0 ± 0.2 mm (length), and 2.1 ± 0.1 mm (thickness), in accordance with ISO 6872:2015 [19] guidelines. These measurements were adjusted based on an estimated 20% shrinkage after sintering.

Specimens were fabricated from pre-sintered multilayer zirconia discs using a precision cutting machine (Secotom 60, Struers, Denmark). Surface polishing was performed using 600- and 1200-grit silicon carbide (SiC) abrasive papers under continuous water irrigation to ensure standardized and smooth surfaces.

The specimens were divided into six experimental groups based on the zirconia type and sintering protocol:

- YML-7 h (YML sintered for 7 hours)
- YML-54 min (YML sintered for 54 minutes)
- YML-51 min (YML sintered for 51 minutes)
- UTML-7 h (UTML sintered for 7 hours)
- UTML-54 min (UTML sintered for 54 minutes)
- UTML-51 min (UTML sintered for 51 minutes)

In each group, 14 specimens were allocated for three-point flexural strength testing, 1 specimen was reserved for scanning electron microscopy (SEM), and 1 for X-ray diffraction (XRD), resulting in a total of 16 specimens per group and 96 specimens overall.

2.3. Sintering Protocols

Three different sintering protocols were applied using a high-speed dental sintering furnace (inFire HTC Speed, Sirona, Bensheim, Germany). The sintering procedures and temperature profiles were selected based on manufacturer recommendations and are detailed as follows:

7-hour conventional sintering protocol

- Heating from room temperature to 1550 °C at 10 °C/min
- Holding at 1550 °C for 2 hours
- Cooling to room temperature at -10 °C/min

54-minute high-speed sintering protocol

- Heating from room temperature to 1450 °C at a rate of 120 °C/min
- Then to 1600 °C at 10 °C/min
- Holding at 1600 °C for 20 minutes
- Cooling to 800 °C at -120 °C/min

51-modified high-speed sintering protocol

- Heating from room temperature to 1400 °C at 50 °C/min
- Then to 1500 °C at 24 °C/min
- Then to 1560 °C at 24 °C/min
- No holding time
- Cooling according to automatic program schedule (manufacturer default)

2.4. Flexural Strength Testing

Three-point flexural strength testing was conducted using a universal testing machine (Lloyd LRX, USA) with a span of 20 mm and crosshead speed of 1 mm/min following ISO 6872 6872:2015.

The flexural strength (σ) was calculated using:

$$\sigma = (3FL) / (2wb^2) \text{ where:}$$

F = fracture load (N), L = span (mm), w = width (mm), b = thickness (mm).

2.5. SEM and Grain Size Analysis

One specimen from each group was sputter-coated with a 3 nm gold layer using a Sputter Coater SC502 (Polaron, VG Microtech, UK) to enhance surface conductivity and was then examined under a field emission scanning electron microscope (FE-SEM; QUANTA 400F, ThermoFisher) at $\times 50,000$ magnifications. Grain size measurements were performed using ImageJ software, with ten randomly selected grains measured per image.

2.6. EDS Analysis

Elemental analysis (Zr, Y, O) was conducted with energy-dispersive spectroscopy (EDS) integrated into the SEM system.

2.7. XRD Analysis

Crystalline phase analysis was performed using a Bruker D8 Advance XRD system (30 mA, 40 kV) with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). The scanning range was 10° – 90° (2θ) at a step size of 0.02° with a speed of $0.6^{\circ}/\text{min}$. Phase identification was performed by comparing the experimental patterns with reference data. Quantitative phase composition was determined using the Whole Powder Pattern Fitting (WPPF) method implemented in Rigaku SmartLab Studio II software, based on pseudo-Voigt profile functions and the PDF-4+ database.

2.8. Statistical Analysis

The flexural strength and grain size data were assessed for normality using the Kolmogorov–Smirnov test. One-way analysis of variance (ANOVA), followed by Tukey’s post hoc test, was used to determine statistically significant differences among groups. A significance level of $p < 0.05$ was applied for all analyses.

3. Results

3.1. Flexural Strength Results

The results of the three-point flexural strength test are summarized in Table 2. In the YML group, the highest flexural strength was observed in the YML--54 min group ($916.7 \pm 209.6 \text{ MPa}$), followed by YML--51 min ($845.0 \pm 249.8 \text{ MPa}$), and YML--7 h ($828.8 \pm 182.4 \text{ MPa}$). Although the difference among sintering durations was not statistically significant ($p > 0.05$), the highest strength was achieved with the 54-minute protocol. In the UTML group, the UTML--51 min subgroup showed the highest strength ($583.7 \pm 108.9 \text{ MPa}$), followed by UTML--54 min ($574.9 \pm 150.7 \text{ MPa}$), and the lowest value was recorded in UTML--7 h ($531.8 \pm 160.3 \text{ MPa}$). Again, no significant difference was found between the sintering durations within the UTML group ($p > 0.05$). When comparing the two materials, YML demonstrated significantly higher flexural strength than UTML in all sintering protocols ($p < 0.05$). Overall, shorter sintering durations (51 min and 54 min) resulted in higher or comparable flexural strength values compared to the conventional 7 h protocol. This trend was more evident in the YML groups. The results of the three-point flexural strength test are summarized in Table 2 and Figure 1.

Table 2. Flexural Strength Values (MPa, Mean $\pm$ SD).

Material	7 h		54 min		51 min		Overall		Time- Based
	(n=28)		(n=28)		(n=28)		(n=86)		
	Mean	SS	Mean	SS	Mean	SS	Mean	SS	
YML (n=42)	828,8	182,4	916,7	209,6	845,0	249,8	863,5	213,9	p** (F)
UTML (n=42)	531,8	160,3	574,9	150,7	583,7	108,9	563,5	140,1	0,741 (-0,30)
Overall (n=84)	680,3	226,4	745,8	249,8	714,3	231,2	713,5	234,7	0,435 (-0,86)
Material based p* (t)	0,00 (-4,83)		0,00 (-4,32)		0,00 (-3,59)		0,00 (-7,45)		0,532 (-0,64)

Student’s t-test, **: ANOVA, a, b, c: Different letters indicate statistically significant differences between columns.

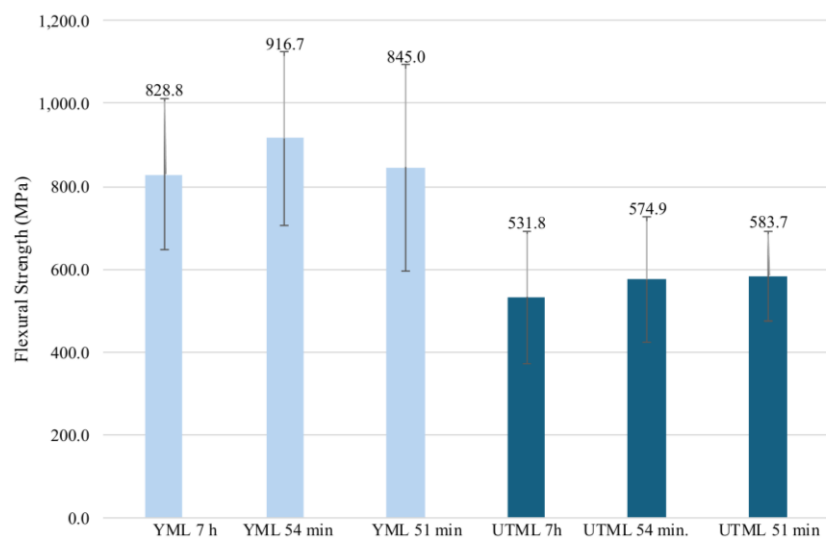


Figure 1. Mean flexural strength (MPa) of YML and UTML groups sintered for 7 h, 54 min, and 51 min. Error bars indicate standard deviation.

3.2. SEM and Grain Size Results

SEM observations revealed distinct microstructural differences among the sintering protocols. Specimens sintered for 54 minutes exhibited the most compact and homogeneous grain structure, followed closely by those sintered for 51 minutes. In contrast, specimens subjected to the 7-hour protocol demonstrated larger, more irregular grain boundaries with occasional voids and porosity.

Grain size measurements for each material group are presented in Table 3. In the YML groups, the 54-minute protocol resulted in the smallest average grain size (234.2 ± 128.4 nm), followed by the 51-minute protocol (406.8 ± 107.0 nm), and the 7-hour group (644.2 ± 146.5 nm). In the UTML groups, the grain sizes were 375.5 ± 116.6 nm (54 min), 386.8 ± 115.5 nm (51 min), and 620.9 ± 80.1 nm (7 h). Statistically significant differences were observed among sintering durations within each material ($p < 0.05$). The results are summarized in Table 3 and Figure 2.

Table 3. Mean Grain Size Values (nm $\pm$ SD).

Material	7 h (n=20)		54 min (n=20)		51 min (n=12)		Overall (n=60)		Time- Based p** (F)
	Mean	SS	Mean	SS	Mean	SS	Mean	SS	
YML (n=30)	^a 644,2	146,5	^b 234,2	128,4	^c 406,8	107,0	428,4	211,1	0,000 (25,75)
UTML (n=30)	^a 620,9	80,1	^b 375,5	116,6	^{bc} 386,8	115,5	461,1	153,6	0,000 (17,21)
Overall (n=60)	^a 632,6	115,5	^b 304,8	139,6	^{bc} 396,8	108,8	444,7	183,8	0,001 (37,63)
Material based p* (t)	0,665 (0,441)		0,019 (-2,578)		0,692 (0,402)		0,496 (-0,686)		

Student’s t-test, **: ANOVA, a, b, c: Different letters indicate statistically significant differences between columns.

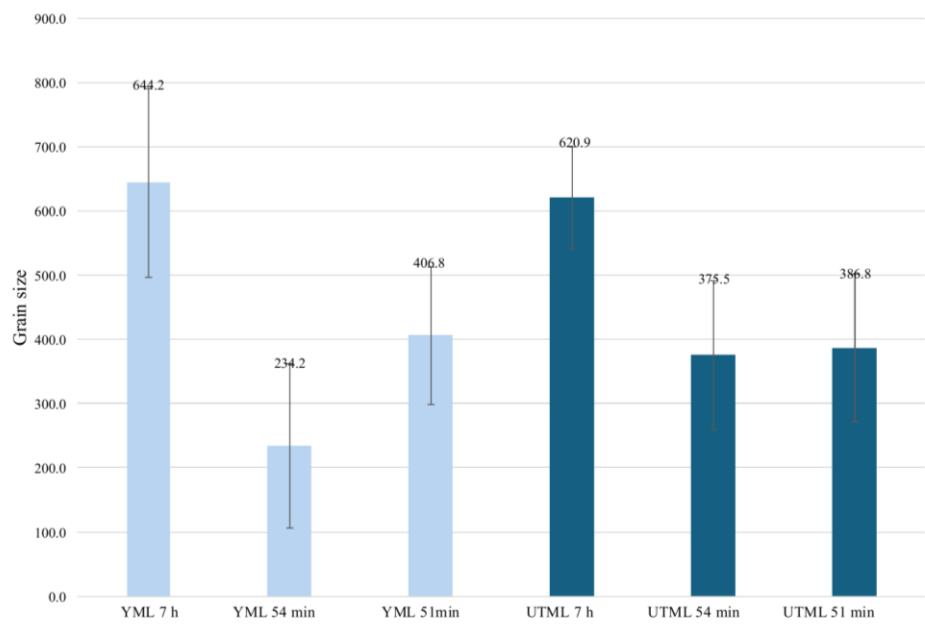


Figure 2. Average grain size (nm) of YML and UTML groups sintered for 7 h, 54 min, and 51 min, based on SEM analysis at 50,000× magnification.

Overall, shorter sintering times resulted in more refined and consistent grain structures. These microstructural advantages observed in the speed sintering groups may contribute to the superior mechanical performance reported in flexural strength results. Representative SEM images for each group are presented in Figures 3 and 4.

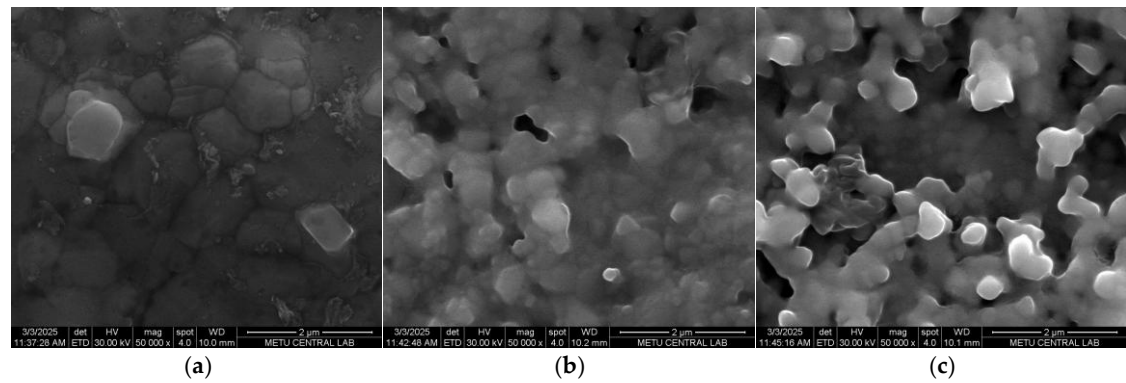


Figure 3. Representative SEM micrographs of YML specimens sintered at different durations, captured at 50,000× magnification: (a) YML-7 h, (b) YML-54 min, (c) YML-51 min.

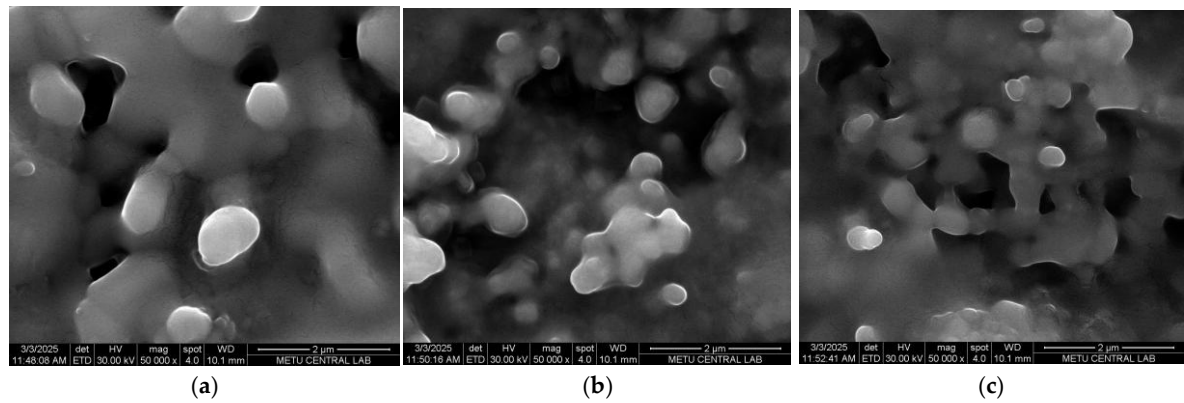


Figure 4. Representative SEM micrographs of UTML specimens sintered at different durations, captured at 50,000× magnification: (a) UTML-7 h, (b) UTML-54 min, (c) UTML-51 min.

3.3. EDS Results

EDS analysis was conducted to evaluate the elemental distribution of zirconium (Zr), yttrium (Y), and oxygen (O) in the YML and UTML specimens across different sintering durations. Table 4 summarizes the mean weight percentages (% wt) of these elements.

In the YML groups, yttrium content ranged from 7.21% (7 h) to 9.56% (54 min). A slight increase in Y content was observed in the speed sintering groups, with values of 9.56% (54 min) and 9.07% (51 min), compared to 7.21% in the 7-hour group. For UTML, the Y content remained more consistent, with 9.97% (7 h), 10.32% (54 min), and 10.27% (51 min). The results are summarized in Table 4.

Table 4. Elemental Composition by EDS (% weight).

Material	Sintering Duration	Zr (%)	Y (%)	O (%)
YML	7 h	79.06	7.21	13.73
YML	54 min	76.51	9.56	13.93
YML	51 min	76.56	9.07	14.37
UTML	7 h	77.43	9.97	12.60
UTML	54 min	75.43	10.32	14.25
UTML	51 min	76.04	10.27	13.69

Zirconium and oxygen contents also showed minor variations, but these were not statistically significant. The overall elemental composition remained within expected ranges for yttria-stabilized zirconia. The speed sintering protocols did not negatively affect elemental stability, as confirmed by EDS spectra.

These findings suggest that shorter sintering durations do not adversely affect the elemental homogeneity of YML or UTML zirconia and may enhance phase consistency by preserving the intended yttria distribution.

3.4. XRD Results

X-ray diffraction (XRD) analysis revealed stable tetragonal–cubic phase compositions across all sintering protocols, with no detectable monoclinic phase. The cubic phase content increased with shorter sintering durations, most prominently in UTML (51 min protocol). Detailed phase fractions for all groups are summarized in Table 5.

Table 5. Phase Composition (% weight) from XRD Analysis.

Material	Sintering Duration	Tetragonal (%)	Cubic (%)
UTML	7 h	23 ± 2	77 ± 2
UTML	54 min	35 ± 2	65 ± 2
UTML	51 min	8 ± 2	92 ± 2
YML	7 h	61.2 ± 0.9	38.8 ± 0.9
YML	54 min	56.3 ± 1.1	43.7 ± 1.1
YML	51 min	46 ± 1	54 ± 1

Representative diffraction patterns are shown in Figures 5–7, with identified phases and corresponding ICDD/PDF card numbers indicated in the captions.

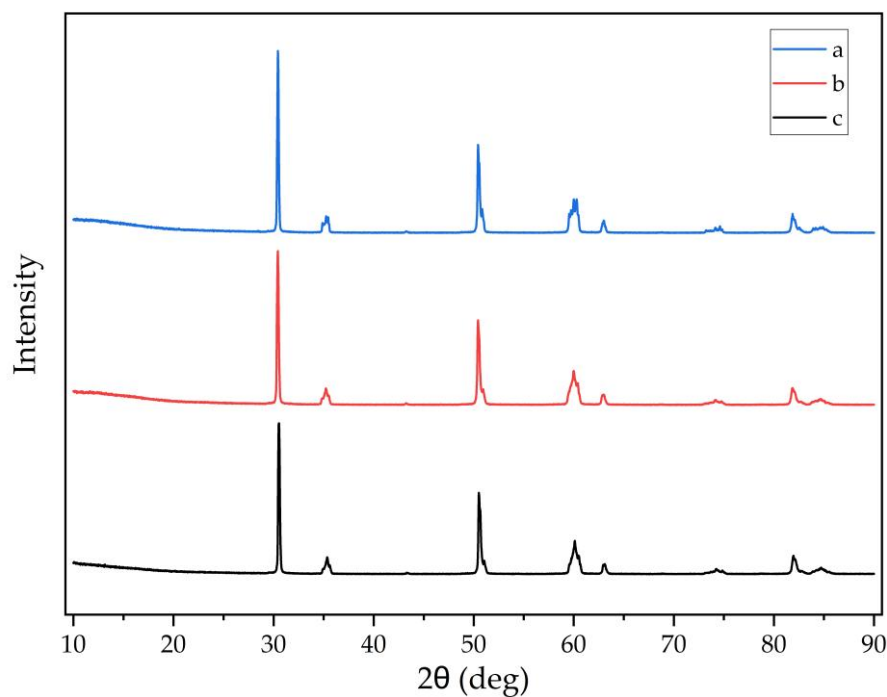


Figure 5. X-ray diffraction (XRD) patterns of YML specimens sintered with different protocols: (a) 7 h at 1550 °C, (b) 54 min at 1600 °C, and (c) 51 min at 1600 °C. The main crystalline phases identified were tetragonal ZrO₂ (P42/nmc; ICDD/PDF 00-050-1089, 01-079-1771) and cubic ZrO₂ (Fm-3m, tazheranite; ICDD/PDF 00-022-0540).

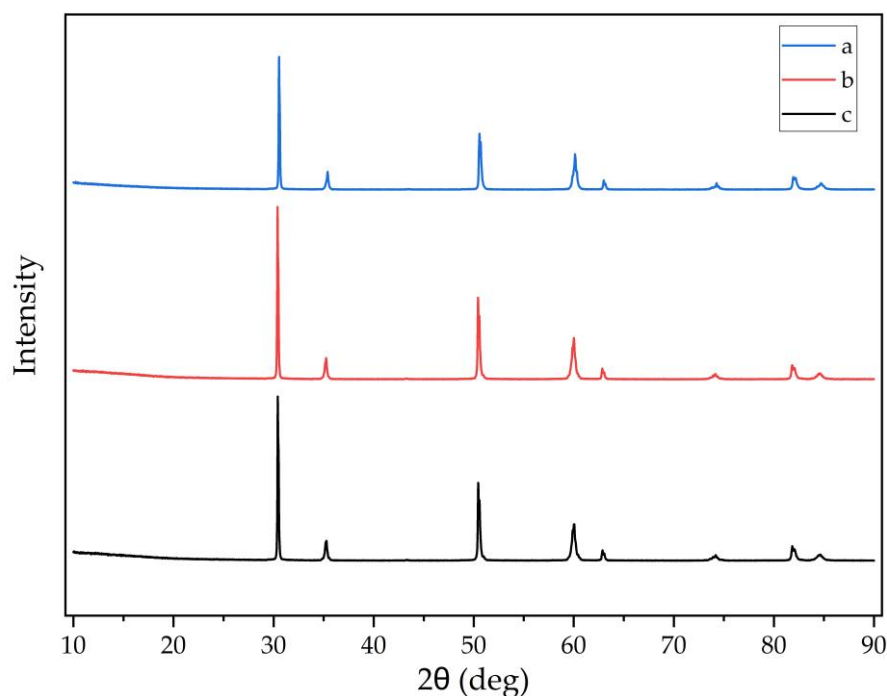


Figure 6. X-ray diffraction (XRD) patterns of UTML specimens sintered with three different protocols: (a) 7 h at 1550 °C, (b) 54 min at 1600 °C, and (c) 51 min at 1600 °C. The main crystalline phases identified were tetragonal ZrO₂ (P42/nmc; ICDD/PDF 01-070-7300, 04-026-7650) and cubic ZrO₂ (Fm-3m, tazheranite; ICDD/PDF 01-078-3193, 04-006-1370).

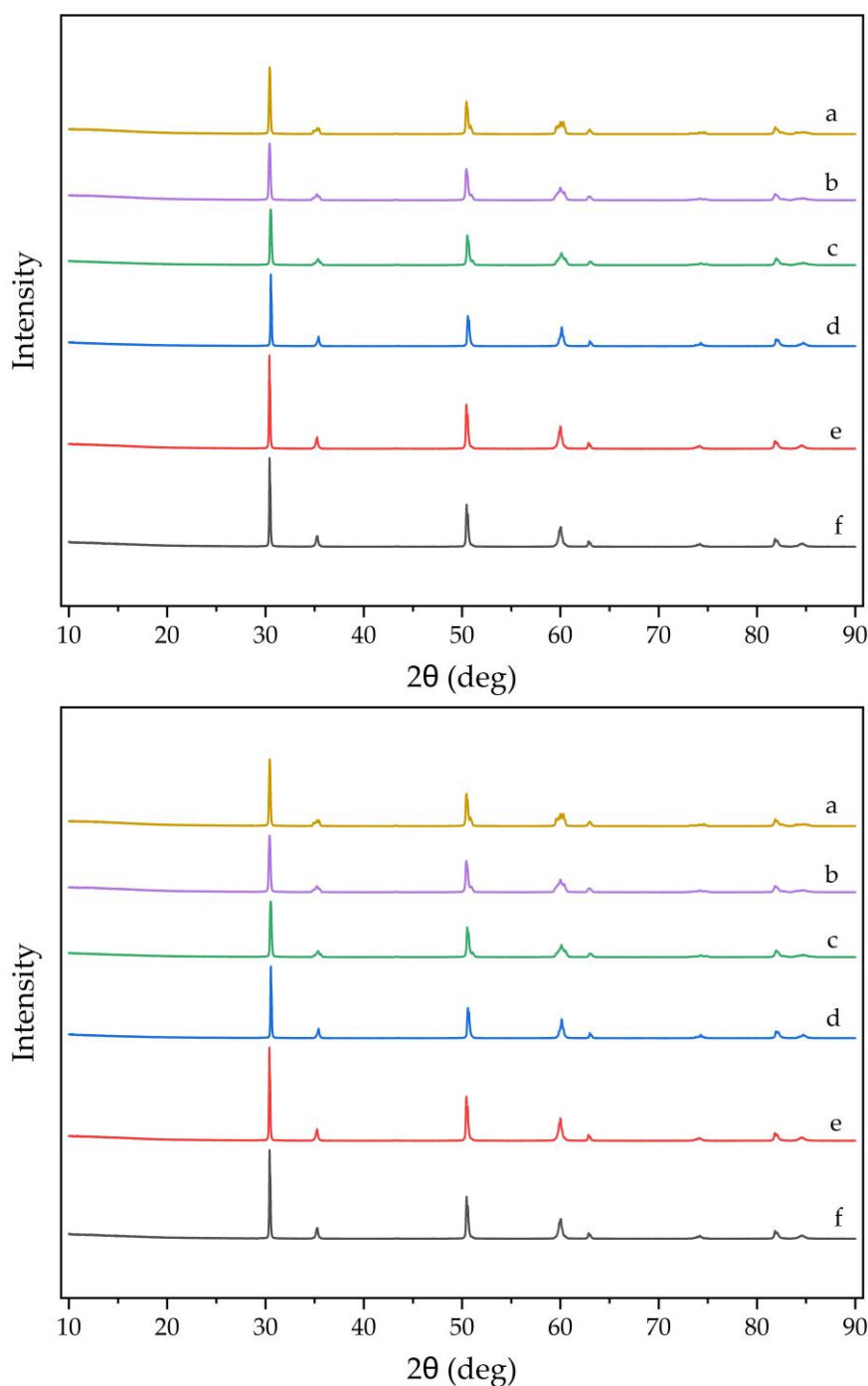


Figure 7. Comparative XRD patterns of YML and UTML specimens under different sintering protocols. (a–c) YML groups: 7 h, 54 min, 51 min; (d–f) UTML groups: 7 h, 54 min, 51 min.

4. Discussion

The present in vitro study investigated the effects of three different sintering protocols: conventional (7 h at 1550 °C), manufacturer-recommended high-speed (54 min at 1600 °C), and an modified high-speed protocol (51 min at 1600 °C) on the flexural strength, microstructure, and phase composition of two multilayer zirconia materials: strength-gradient KATANA™ YML and color-gradient KATANA™ UTML. A total of 96 bar-shaped specimens were prepared and analyzed using mechanical testing, scanning electron microscopy (SEM), energy-dispersive spectroscopy (EDS), and X-ray diffraction (XRD). The null hypothesis proposed that reducing sintering time would not significantly alter the mechanical and microstructural properties of these zirconia materials. Based on the findings, this hypothesis was partially accepted: sintering duration did not significantly affect

flexural strength within each material group, but it did influence grain size and phase composition. Flexural strength testing showed that YML consistently outperformed UTML in all sintering protocols, with the highest strength recorded for YML after 54 minutes of sintering. In UTML, the 51-minute protocol yielded the greatest strength, although differences between protocols were not statistically significant. These results indicate that accelerated sintering can produce mechanical performance equal to or greater than conventional sintering.

Previous studies have reported inconsistent findings regarding the relationship between sintering temperature, duration, and zirconia strength. Some investigations indicated that increasing sintering temperature and time did not produce significant changes in hardness or flexural strength, though improvements in translucency and shade matching were observed [20]. Others found that grain size in 3Y-TZP increased sharply when temperatures exceeded 1300 °C, with the largest grains at 1700 °C, which was associated with a decline in flexural strength and optical contrast [15]. In contrast, some reports suggested that combining high temperatures with short sintering times could enhance flexural strength, although microstructural evidence was limited [12]. The present results are consistent with the latter, as both accelerated protocols (51 and 54 min) produced equal or higher flexural strength compared with the conventional 7-hour process, accompanied by finer and more homogeneous microstructures. Attia et al.[16] similarly reported that high-speed sintering improved fracture strength and reduced grain size in gradient zirconia fixed partial dentures, with a negative correlation observed between grain size and strength. These outcomes align with the present study, supporting the concept that short-duration, high-temperature sintering improves zirconia's mechanical behavior by restricting grain growth.

In this study, the higher flexural strength in accelerated sintering groups may be related to finer and more homogeneous grains. Elevated temperatures (1600 °C) promote particle coalescence, reduce porosity, and increase density [12,17]. These results agree with studies showing that high-speed sintering or short durations at high temperatures enhance flexural strength [12,21]. By contrast, the lower strength in conventionally sintered specimens may reflect larger grains formed during prolonged sintering and holding [22–24], which can promote grain growth, tetragonal-to-monoclinic transformation, and yttria migration [15,25,26]. The small variations observed are thus more likely linked to grain size consistent with some reports [25,27], though others found no significant differences between protocols [28–30] or suggested strength is unaffected by material, sintering time, or temperature [10,11,20,31,32]. Grain size analysis confirmed that accelerated sintering produced finer microstructures than conventional sintering. In YML, the 54-minute group showed the smallest mean grain size (234.2 ± 128.4 nm), followed by the 51-minute group (406.8 ± 107.0 nm), while the 7-hour group exhibited larger grains (644.2 ± 146.5 nm). In UTML, values were 375.5 ± 116.6 nm (54 min), 386.8 ± 115.5 nm (51 min), and 620.9 ± 80.1 nm (7 h). All values remained below the critical threshold of 1 μ m, above which tetragonal-to-monoclinic transformation may occur [26] and none approached the 0.2 μ m limit associated with loss of transformation toughening [10]. These findings confirm that sintering strongly influences grain size [17,33], and that shorter durations effectively limited grain growth without adverse microstructural changes. Regardless of the protocol, few studies have evaluated the mechanical performance of gradient zirconia, and direct comparisons remain difficult due to differences in material composition and experimental design [31,34–36]. In this study, YML consistently showed higher flexural strength than UTML, which can be explained by its design: YML contains regions with lower yttria content that stabilize the tetragonal phase and promote transformation toughening, while UTML's uniformly higher yttria favors translucency but reduces strength. EDS confirmed stable composition overall, with only a slight increase in Y content in accelerated YML groups (7.21% $\rightarrow$ 9.56%), whereas UTML remained unchanged ($\approx$ 10%). Previous reports also emphasize the role of yttria distribution and layer-specific properties, noting that YML's body layers (3–5 mol% Y_2O_3) provide higher strength than transition layers [18,37]. Although higher yttria has been linked to larger grains and reduced strength [38], YML has still been shown to outperform other multilayer zirconias [18,37]. Moreover, evidence that repeated or high-speed

sintering does not reduce biaxial strength[23,39] supports the present results that accelerated sintering preserved mechanical performance.

XRD analysis confirmed the absence of monoclinic phase in all groups, indicating stable phase composition across protocols. As expected from its higher yttria content, UTML exhibited a cubic-dominant structure under all conditions. In YML, which contains less yttria in load-bearing regions, the cubic phase fraction increased with accelerated sintering (38.8% at 7 h → 43.7% at 54 min → 54% at 51 min), accompanied by a decrease in tetragonal content. Although this change did not significantly affect flexural strength, the higher tetragonal proportion in YML compared with UTML may explain its superior performance. The toughness of the tetragonal phase is associated with transformation toughening, where localized tetragonal-to-monoclinic transformation produces compressive stresses that inhibit crack propagation[6,9]. These findings are consistent with previous reports showing that 3Y-TZP regions are more prone to phase transformation during sintering, whereas higher yttria (5Y-TZP) provides greater stability through diffusion and cubic phase stabilization[11,37]. This explains why YML exhibited protocol-dependent tetragonal–cubic variations, while UTML consistently maintained a cubic structure. Güntekin[40], similarly reported that cubic content increased with high-speed and repeated sintering, particularly in YML, and that repeated firings reduced differences between conventional and accelerated groups. Overall, these results indicate that phase ratios are primarily influenced by yttria content and sintering history, while mechanical behavior is more strongly governed by grain size refinement [41]. Recent work by Nonaka et al. [42] demonstrated that rapid cooling in dental zirconia generates a compressive residual stress layer on the surface, which increases in thickness with faster cooling rates. Although flexural strength was not significantly affected, fracture toughness was improved due to suppression of crack propagation by this stress layer. This supports the possibility that, in addition to grain refinement (Hall–Petch effect)[41], residual stresses induced by the fast-cooling rate in high-speed sintering may also contribute to the mechanical performance of zirconia ceramics.

Our XRD results showed that all sintering protocols prevented the formation of monoclinic phases, while variations in tetragonal-to-cubic ratios were observed depending on the cycle. These findings are consistent with previous work on YSZ ceramics by Lis et al.[43], who demonstrated that sintering temperature critically influences phase stability. In their study, 6YSZ ceramics sintered at 1550 °C showed the maximum tetragonal fraction (≈56 wt%) and improved fracture toughness, while further temperature increases to 1600 °C promoted grain growth and a shift toward monoclinic transformation, leading to reduced mechanical properties. In contrast, our multilayered zirconia specimens did not exhibit monoclinic formation even at 1600 °C cycles, likely due to their higher yttria content and compositional gradient design. Nevertheless, both studies underline that the balance between tetragonal and cubic phases plays a decisive role in mechanical behavior. While Lis et al.[43] highlighted temperature-driven effects, our results further indicate that sintering time is equally critical, as ultrafast cycles promoted cubic stabilization without compromising strength.

This study was conducted under in vitro conditions and did not replicate the complex variables of the oral environment, such as thermal cycling, pH fluctuations, and long-term mechanical fatigue. Only two multilayer zirconia materials and three sintering protocols were evaluated, which may limit the generalizability of the results. Optical properties were not assessed, so the esthetic consequences of the observed microstructural and phase changes remain unclear. Additionally, the study focused solely on flexural strength, and other mechanical parameters, such as fracture toughness and wear resistance, should be addressed in future research.

5. Conclusions

Shortening the sintering time to 54 or 51 minutes did not significantly affect the flexural strength of multilayered monolithic zirconia materials compared to conventional 7-hour sintering.

High-speed sintering protocols produced smaller and more homogeneous grain structures, especially in the YML groups.

Both zirconia materials-maintained phase stability under all sintering conditions. No monoclinic phase was detected.

YML consistently showed higher flexural strength and more favorable microstructural characteristics than UTML, regardless of the sintering duration.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org.

Author Contributions: Lana Alatrash: Conceptualization, Methodology, Investigation, Data curation, Writing – original draft. Asude Dilek Nalbant: Supervision, Validation, Resources, Writing – review & editing.

Funding: This study was supported by the Scientific Research Projects Coordination Unit of Gazi University under project number TDH-2024-9487.

Institutional Review Board Statement: Not applicable. This in vitro study involved no human participants, animals, or personal data and therefore did not require ethical approval in accordance with local legislation and institutional guidelines.

Informed Consent Statement: Not applicable.

Data Availability Statement: The data supporting the findings of this study are available from the corresponding author upon reasonable request. No publicly archived datasets were used or generated.

Acknowledgments: This study was supported by the Scientific Research Projects Coordination Unit of Gazi University under project number **TDH-2024-9487**. The authors would like to express their sincere gratitude to Gazi University for providing financial and infrastructural support throughout the research process.

Conflicts of Interest: The authors declare no conflicts of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

References

1. Spitznagel FA, Boldt J, Gierthmuehlen PC. CAD/CAM Ceramic Restorative Materials for Natural Teeth. *J Dent Res* 2018;97:1082–91. <https://doi.org/10.1177/0022034518779759>.
2. Alghazzawi TF. Advancements in CAD/CAM technology: Options for practical implementation. *J Prosthodont Res* 2016;60:72–84. <https://doi.org/10.1016/J.JPOR.2016.01.003>.
3. Kelly JR, Denry I. Stabilized zirconia as a structural ceramic: An overview. *Dental Materials* 2008;24:289–98. <https://doi.org/10.1016/J.DENTAL.2007.05.005>.
4. Piconi C, Maccauro G. Zirconia as a ceramic biomaterial. *Biomaterials* 1999;20:1–25. [https://doi.org/10.1016/S0142-9612\(98\)00010-6](https://doi.org/10.1016/S0142-9612(98)00010-6).
5. Leib EW, Vainio U, Pasquarelli RM, Kus J, Czacshke C, Walter N, et al. Synthesis and thermal stability of zirconia and yttria-stabilized zirconia microspheres. *J Colloid Interface Sci* 2015;448:582–92. <https://doi.org/10.1016/J.JCIS.2015.02.049>.
6. Kongkiatkamon S, Rokaya D, Kengtanyakich S, Peampring C. Current classification of zirconia in dentistry: an updated review. *PeerJ* 2023;11:e15669. <https://doi.org/10.7717/PEERJ.15669>.
7. Hannink RHJ, Kelly PM, Muddle BC. Transformation toughening in zirconia-containing ceramics. *Journal of the American Ceramic Society* 2000;83:461–87. <https://doi.org/10.1111/J.1151-2916.2000.TB01221.X>.
8. Lim CH, Vardhaman S, Reddy N, Zhang Y. Composition, processing, and properties of biphasic zirconia bioceramics: Relationship to competing strength and optical properties. *Ceram Int* 2022;48:17095–103. <https://doi.org/10.1016/j.ceramint.2022.02.265>.
9. Sulaiman TA, Suliman AA, Abdulmajeed AA, Zhang Y. Zirconia restoration types, properties, tooth preparation design, and bonding. A narrative review. *Journal of Esthetic and Restorative Dentistry* 2024;36:78–84. <https://doi.org/10.1111/JERD.13151>.
10. Öztürk C, Can G. Effect of Sintering Parameters on the Mechanical Properties of Monolithic Zirconia. *J Dent Res Dent Clin Dent Prospects* 2019;13:247–52. <https://doi.org/10.15171/JODDD.2019.038>.

11. Cokic SM, Vleugels J, Van Meerbeek B, Camargo B, Willems E, Li M, et al. Mechanical properties, aging stability and translucency of speed-sintered zirconia for chairside restorations. *Dent Mater* 2020;36:959–72. <https://doi.org/10.1016/J.DENTAL.2020.04.026>.
12. Ersoy NM, Aydoğdu HM, Değirmenci BÜ, Çökük N, Sevimay M. The effects of sintering temperature and duration on the flexural strength and grain size of zirconia. *Acta Biomater Odontol Scand* 2015;1:43–50. <https://doi.org/10.3109/23337931.2015.1068126>.
13. Kaizer MR, Gierthmuehlen PC, dos Santos MB, Cava SS, Zhang Y. Speed sintering translucent zirconia for chairside one-visit dental restorations: Optical, mechanical, and wear characteristics. *Ceram Int* 2017;43:10999–1005. <https://doi.org/10.1016/J.CERAMINT.2017.05.141>.
14. Juntavee N, Attashu S. Effect of different sintering process on flexural strength of translucency monolithic zirconia. *J Clin Exp Dent* 2018;10:e821–30. <https://doi.org/10.4317/JCED.54749>.
15. Stawarczyk B, Özcan M, Hallmann L, Ender A, Mehl A, Hämmerlet CHF. The effect of zirconia sintering temperature on flexural strength, grain size, and contrast ratio. *Clin Oral Investig* 2013;17:269–74. <https://doi.org/10.1007/S00784-012-0692-6>.
16. Attia MA, Radwan M, Blunt L, Bills P, Tawfik A, Arafa AM. Effect of different sintering protocols on the fracture strength of 3-unit monolithic gradient zirconia fixed partial dentures: An in vitro study. *Journal of Prosthetic Dentistry* 2023;130:908.e1–908.e8. <https://doi.org/10.1016/j.prosdent.2023.09.012>.
17. Gómez SY, Da Silva AL, Gouvêa D, Castro RHR, Hotza D. Nanocrystalline yttria-doped zirconia sintered by fast firing. *Mater Lett* 2016;166:196–200. <https://doi.org/10.1016/J.MATLET.2015.12.042>.
18. Strasser T, Wertz M, Koenig A, Koetzsche T, Rosentritt M. Microstructure, composition, and flexural strength of different layers within zirconia materials with strength gradient. *Dental Materials* 2023;39:463–8. <https://doi.org/10.1016/j.dental.2023.03.012>.
19. ISO 6872:2015 - Dentistry — Ceramic materials n.d. <https://www.iso.org/standard/59936.html> (accessed February 23, 2025).
20. Ebeid K, Wille S, Hamdy A, Salah T, El-Etreby A, Kern M. Effect of changes in sintering parameters on monolithic translucent zirconia. *Dental Materials* 2014;30:e419–24. <https://doi.org/10.1016/J.DENTAL.2014.09.003>.
21. Zimmermann M, Ender A, Mehl A. Influence of CAD/CAM Fabrication and Sintering Procedures on the Fracture Load of Full-Contour Monolithic Zirconia Crowns as a Function of Material Thickness. *Oper Dent* 2020;45:219–26. <https://doi.org/10.2341/19-086-L>.
22. Kim MJ, Ahn JS, Kim JH, Kim HY, Kim WC. Effects of the sintering conditions of dental zirconia ceramics on the grain size and translucency. *J Adv Prosthodont* 2013;5:161. <https://doi.org/10.4047/JAP.2013.5.2.161>.
23. Liu H, Inokoshi M, Nozaki K, Shimizubata M, Nakai H, Cho Too T Di, et al. Influence of high-speed sintering protocols on translucency, mechanical properties, microstructure, crystallography, and low-temperature degradation of highly translucent zirconia. *Dental Materials* 2022;38:451–68. <https://doi.org/10.1016/J.DENTAL.2021.12.028>.
24. Attachoo S, Juntavee N. Role of sintered temperature and sintering time on spectral translucence of nano-crystal monolithic zirconia. *J Clin Exp Dent* 2019;11:e146–53. <https://doi.org/10.4317/JCED.55497>.
25. Mayinger F, Pfefferle R, Reichert A, Stawarczyk B. Impact of High-Speed Sintering of Three-Unit 3Y-TZP and 4Y-TZP Fixed Dental Prostheses on Fracture Load With and Without Artificial Aging. *Int J Prosthodont* 2021;34:47–53. <https://doi.org/10.11607/IJP.6775>.
26. Bravo-Leon A, Morikawa Y, Kawahara M, Mayo MJ. Fracture toughness of nanocrystalline tetragonal zirconia with low yttria content. *Acta Mater* 2002;50:4555–62. [https://doi.org/10.1016/S1359-6454\(02\)00283-5](https://doi.org/10.1016/S1359-6454(02)00283-5).
27. Inokoshi M, Zhang F, De Munck J, Minakuchi S, Naert I, Vleugels J, et al. Influence of sintering conditions on low-temperature degradation of dental zirconia. *Dent Mater* 2014;30:669–78. <https://doi.org/10.1016/J.DENTAL.2014.03.005>.
28. Ahmed WM, Troczynski T, McCullagh AP, Wyatt CCL, Carvalho RM. The influence of altering sintering protocols on the optical and mechanical properties of zirconia: A review. *J Esthet Restor Dent* 2019;31:423–30. <https://doi.org/10.1111/JERD.12492>.

29. Jansen JU, Lümekemann N, Letz I, Pfefferle R, Sener B, Stawarczyk B. Impact of high-speed sintering on translucency, phase content, grain sizes, and flexural strength of 3Y-TZP and 4Y-TZP zirconia materials. *Journal of Prosthetic Dentistry* 2019;122:396–403. <https://doi.org/10.1016/j.prosdent.2019.02.005>.
30. Elisa Kauling A, Güth JF, Erdelt K, Edelhoff D, Keul C. Influence of speed sintering on the fit and fracture strength of 3-unit monolithic zirconia fixed partial dentures. *J Prosthet Dent* 2020;124:380–6. <https://doi.org/10.1016/J.PROSDENT.2019.09.003>.
31. Michailova M, Elsayed A, Fabel G, Edelhoff D, Zylla IM, Stawarczyk B. Comparison between novel strength-gradient and color-gradient multilayered zirconia using conventional and high-speed sintering. *J Mech Behav Biomed Mater* 2020;111. <https://doi.org/10.1016/J.JMBBM.2020.103977>.
32. Hjerpe J, Närhi T, Fröberg K, Vallittu PK, Lassila LVJ. Effect of shading the zirconia framework on biaxial strength and surface microhardness. *Acta Odontol Scand* 2008;66:262–7. <https://doi.org/10.1080/00016350802247123>.
33. Kim MJ, Ahn JS, Kim JH, Kim HY, Kim WC. Effects of the sintering conditions of dental zirconia ceramics on the grain size and translucency. *Journal of Advanced Prosthodontics* 2013;5:161–6. <https://doi.org/10.4047/JAP.2013.5.2.161>.
34. Oyar P, Durkan R, Deste G. Effects of sintering time and hydrothermal aging on the mechanical properties of monolithic zirconia ceramic systems. *Journal of Prosthetic Dentistry* 2021;126:688–91. <https://doi.org/10.1016/J.PROSDENT.2020.09.006>.
35. Rosentritt M, Preis V, Schmid A, Strasser T. Multilayer zirconia: Influence of positioning within blank and sintering conditions on the in vitro performance of 3-unit fixed partial dentures. *J Prosthet Dent* 2022;127:141–5. <https://doi.org/10.1016/J.PROSDENT.2020.11.009>.
36. Attia A, Kern M. Influence of cyclic loading and luting agents on the fracture load of two all-ceramic crown systems. *J Prosthet Dent* 2004;92:551–6. <https://doi.org/10.1016/J.PROSDENT.2004.09.002>.
37. Inokoshi M, Liu H, Yoshihara K, Yamamoto M, Tonprasong W, Benino Y, et al. Layer characteristics in strength-gradient multilayered yttria-stabilized zirconia. *Dent Mater* 2023;39:430–41. <https://doi.org/10.1016/J.DENTAL.2023.03.015>.
38. Pekkan G, Pekkan K, Bayindir BÇ, Özcan M, Karasu B. Factors affecting the translucency of monolithic zirconia ceramics: A review from materials science perspective. *Dent Mater J* 2020;39:1–8. <https://doi.org/10.4012/DMJ.2019-098>.
39. Katada H, Inokoshi M, Kamijo S, Liu H, Xu K, Kawashita M, et al. Effects of multiple firings on the translucency, crystalline phase, and mechanical strength of highly translucent zirconia. *Dent Mater J* 2024;43:294–302. <https://doi.org/10.4012/DMJ.2023-255>.
40. Güntekin N, Kızıllırmak B, Tunçdemir AR. Comparison of Mechanical and Optical Properties of Multilayer Zirconia After High-Speed and Repeated Sintering. *Materials* 2025;18. <https://doi.org/10.3390/MA18071493>.
41. Novitskaya E, Karandikar K, Cummings K, Mecartney M, Graeve OA. Hall–Petch effect in binary and ternary alumina / zirconia / spinel composites. *Journal of Materials Research and Technology* 2021;11:823–32. <https://doi.org/10.1016/J.JMRT.2021.01.058>.
42. Nonaka K, Teramae M, Pezzotti G. Effect of rapid cooling on residual stress and surface fracture toughness of dental zirconia. *J Mech Behav Biomed Mater* 2024;157:106656. <https://doi.org/10.1016/J.JMBBM.2024.106656>.
43. Kulyk V, Duriagina Z, Vasylyv B, Kovbasiuk T, Lyutyy P, Vira V, et al. ACTA PHYSICA POLONICA A Effect of Sintering Temperature on Crack Growth Resistance Characteristics of Yttria-Stabilized Zirconia 2022;141. <https://doi.org/10.12693/APhysPolA.141.323>.

Disclaimer/Publisher's Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.