ASDJ
AINSHAMS DENTAL JOURNAL

Print ISSN 1110-7642
Online ISSN 2735-5039

AIN SHAMS DENTAL JOURNAL
Official Publication of Ain Shams Dental School
June 2024 • Vol. 34

The effect of speed sintering and hydrothermal aging on the biaxial flexural strength and microstructure of recent cubic zirconia material

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Aim: To evaluate the effect of speed sintering protocol and hydrothermal aging on the biaxial flexural strength and microstructure of recent cubic zirconia material.

Materials and methods: Discs were prepared from recent cubic zirconia material, Katana UTML (K). The prepared discs were fabricated from milled cylinders. Prepared discs were divided according to sintering protocol applied, either a normal cycle (N) or a speed cycle (S). Half of the discs were subjected to biaxial flexural strength (BFS) test using a universal testing machine. Remaining discs were submitted to accelerated aging (A) for 5 hours and then subjected to biaxial flexural strength (BFS) test. Data collected and analyzed using one-way ANOVA. Scanning electron microscope was used for microstructure analysis.

Results: Non-significant difference in (BFS) values were found between both normal and speed sintering protocols before and after subjecting to hydrothermal aging, The highest overall values were reported from KSA (2168± 212.4 MPa) . Average mean grain size values showed a reduction in size after subjecting to speed sintering protocol.

Conclusion: Speed sintering protocols and hydrothermal aging did not report a significant difference in biaxial flexural strength mean values.

Keywords: speed sintering, cubic zirconia, hydrothermal aging, flexural strength.

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Introduction

Due to the growing demand for aesthetically pleasing and natural looking dental materials, the popularity of all ceramic materials has increased. In the middle of the different All-ceramic dental restorative materials, Yttria-stabilized tetragonal (Y-TZP) zirconia gained its popularity due to its appealing and exceptional aesthetics as well as mechanical properties, in addition to its favorable biocompatibility. [1-3] It is worth noting that the continuous progress in ceramic materials fabrications and microstructure enabled the production of all-ceramic materials with exceptional and superb clinical performance levels, that securing a superlative performance under different challenging clinical situations, covering both the mechanical as well as the optical properties.

Zirconia's first generation with 3 mol% yttria offers a superior mechanical property, where the major drawback about using the Zirconia's first generation was its remarked opacity, so the native intent for using was for frameworks for bridges and crowns. [4] Yttria-stabilized tetragonal original colour of ranging from white opaque to ivory, as a result of its opaque colour Y-TZP should be veneered with translucent feldspathic porcelain like that used with porcelain fused to metal restorations. However, the frequent reason of failure was cohesive failure or chipping of the porcelains layer while the core material, 3Y-TZP remains unaffected, this led to the introduction of what is known as monolithic dental zirconia that expanded the use to offer a full anatomical or full contoured restoration. [5,6]

To facilitate the production of what is known as monolithic dental zirconia with accepted translucency, fabrication of the recent generation of the Yttria-stabilized tetragonal zirconia (3Y-TZPs) was improved by substantially decreasing alumina additive concentrations and reducing porosity by using a higher sintering temperature, which actually resulted in a slight improvement if the optical properties. this generation of 3Y-TZPs could not fulfill the esthetic demands to be used in a full anatomical state for anterior restorations.[7] It is worth mentioning that the concentration of yttria has been reported to affect crucially on zirconia physical properties. [8-10] So, the next generations in monolithic zirconia contained a transparent phase in its final product in order to reduce opacity That was fabricated by increasing the yttria content to produce a partially stabilized zirconia 4mol% (4YPSZ) or 5mol% (4-YPSZ). [11] While this modification improved translucency and optical properties, strength and toughness were negatively affected, as known that cubic zirconia does not experience stress-influenced tetragonal to monoclinic phase transformation.

The concept of monolithic or full countered zirconia materials has expanded to offer a multilayered blanks. This technology imitates the natural teeth color graduation by using different color pigments, with corresponding strength to enamel and dentin.[12-16] Multi-layered zirconia ceramics have raised from the idea of production recent zirconia blanks containing various layers with different and variable shades as well translucencies. [17] The blank layout aims to impersonate the natural teeth shade gradient, where the incisal area is translucent showing an enamel-like appearance and increasing in chroma till reaching the gingival region. [18] The equivalent and even multi-chromatic blanks could be fabricated using a particular approach where zirconia powders are coated with coloring substrates.[19]

Katana Zirconia UTML represents the concept of multilayered monolithic zirconia. According to the manufacturer, Katana Zirconia UTML is contraindicated to be used for a fixed partial denture with three units or longer, and also a minimum required thickness of 0.8 mm is recommended for sufficient strength, but this may compromise the translucency. With respect to the restoration type, UTML is aimed to have minimal thicknesses: 1 mm used for full-coverage posterior units and 0.8 mm for anterior single crowns, and 0.4 mm for veneers. [20]

Speed sintering protocols have been introduced mainly to overcome the main draw backs of using the long conventional cycles that consumes time and energy. [21,22] It is assumed that speed or rapid cycles could result in a more uniform microstructure with optimum density for cubic zirconia. [23] Altering sintering conditions may result in a negative effect on the flexural strength [24-27] or did not show a clear impact, [28,29] where applying different sintering parameters or protocols did not report a significant effect on strength of different zirconia materials.

The impact of low temperature degradation (LTD) on zirconia microstructure and strength is important for restorations longevity. Tetragonal (3Y-TZP) undergo a tetragonal to monoclinic phase transformation as a result of low temperature degradation. [30] while the effect of aging is clear regarding tetragonal zirconia, the effect on cubic (5Y-TZP) needs to be evaluated, as the dominance composition is cubic phase unlike tetragonal phase in (3Y-TZP).

For long term clinical success for brittle materials such as zirconia, fracture strength and fracture toughness [28] considered to be crucial factors, as zirconia materials provided with high strength values are less receptive to fracture. [29] So, the purpose of this study was to assess the effect of speed sintering cycles and hydrothermal aging on the BFS and microstructure of cubic zirconia. while some studies reported a significant effect of sintering cycles on flexural strength (BFS) [21,29,31-33], other studies revealed no different in strength values. [28,29,32,33] The null hypotheses were that speed sintering and hydrothermal aging would not affect the flexural strength and microstructure of cubic zirconia materials.

Materials and methods

Sample size calculation was based on a continuous response variable derived from matched pairs in a prior study by Sadek[34] 2023, it was determined that 11 samples in each group would be adequate to get a large effect size (d = 1.4663818), achieving an actual power (1-β error) of 0.95 (95%) and a significance level (α error) of 0.05 (5%) for a two-sided hypothesis test.

A total of 44 discs were prepared from katana UTML, details are shown in table (1). Discs were divided into two main groups (n=22), normal and speed cycles. Each group was divided into two subgroups (n=11) either no aging subgroup or aging subgroup.

Table (1): katana UTML technical data

Trade name	Chemical composition	Manufacturer	Lot #
KATANA Zirconia UTML	ZrO_2 + HfO 87 – 92 %2, Y_2O_3 8 – 11 % Other oxides 0 – 2 %	Kuraray Noritake	DZMN

A total 10 cylinders from katana UTML were fabricated using 5-axis CNC milling machine (core Tec 250i dry, Germany). Cylinders were created with 14.88 diameter about 20% larger diameter than the desired final dimensions. Discs were fabricated from the cylinders by sectioning the cylinders among its length using (Isomet 4000, Buehler, Lakebluff, U.S.A) under water irrigation. Zirconia discs had a dimensions of 1.25 mm thickness, 20% larger than the intended final dimension to compensate for the sintering shrinkage.

The two sintering cycles (normal and speed) were performed using inFire HTC Speed furnace (Dentsply Sirona, Bensheim, Germany). Discs were sintered according to the desired sintering parameters presented in table (2). After sintering discs reached 12 mm diameter and 1 mm thickness and confirmed using a digital caliper. For Aging protocol discs were subjected to hydrothermal aging in autoclave at 134 °C under a standard H_2O vapor pressure of 2 bar (according to ISO 13356) for 5 hrs. [35,36]

Table (2): Sintering Programs for KATANA™ Zirconia UTML

Sintering protocol	Heating rate	Sintering temperature	Holding time	Cooling rate
Normal cycle	10 °C/min	1550 °C	2 hrs	-10 °C/min
Speed cycle	35 °C/min	1560 °C	0.5 hrs	-45 °C/min

Biaxial flexural strength (BFS) test was performed with a universal testing machine (Instron, university Ave, Norwood, USA). The load was applied at a crosshead constant speed of 1.0 mm/min till fracture. Flexural strength test was performed with and without aging.

Scanning Electron Microscope (supra 55, carl-zeis, oberkochen, Germany) was used to evalute microstructer and grain size before and after hydrothermal aging. A gold sputter coater (3 minute/15 mA) was used to apply a conductive layer prior to imaging to enhance image quality. Grain size assessment was performed using (Image-J software, online version), grains were measured from its boundaries using the analyzing tools then statistically analyzed to conclude the mean and the standard deviation.

Results

The results are presented in two tables: Table (3) provides descriptive statistics for the flexural strength measurements, while Table (4) presents comparative statistics using Student's t-test and paired t-test. Results reported that discs subjected to normal cycle (KN and KNA) in general showed a higher BFS values compared to those values reported from speed sintering (KS and KSA). The maximum BFS values were (2538 MPa for KN and 2267 MPa for KNA), while the maximum values reported for (KS and KSA were lower at 2470 MPa and 2416 Mpa) respectively. Non-significant different was reported between all groups, while the highest overall BFS values were reported from (KSA, 2168± 212.4Mpa). Regarding discs not subjected to aging a higher BFS values was reported from normal cycle (KN, 2162±406.0 MPa), where speed cycle showed (KS, 2130±456.5 MPa). While discs underwent aging showed that speed cycle had a higher BFS values (KSA, 2168±212.4 MPa) compared to values obtained from normal cycle (KNA, 2111.11±118.4 MPa). Scanning electron microscope (SEM) analysis using (Image-J) indicated that the average grain size was reduced with speed sintering shown in table (4). Moreover, aging did not report to have a clear impact in microstructure.

Table (3): Descriptive Statistics of Sintering Cycles Effect on Samples Flexural Strength (MPa) of (Katana UTML Zirconia):

	KN	KNA	KS	KSA
Minimum	1431	1928	1233	1806
25% Percentile	1850	2016	1919	2030
Median	2291	2115	2270	2171
75% Percentile	2460	2213	2409	2358
Maximum	2538	2267	2470	2416
Range	1107	338.8	1237	610.0
Mean	2162	2111	2130	2168
Std. Deviation	406.0	118.4	456.5	212.4
Std. Error of Mean	165.7	48.35	186.4	86.73
Lower 95% CI of mean	1736	1987	1651	1945
Upper 95% CI of mean	2588	2235	2609	2391

Table (4): Comparative Statistics of Sintering Cycles Effect on Samples Flexural Strength (MPa) of (Katana UTMLZirconia)

			Normal Sintering (N) Mean ± SD (95% CI)	Speed Sintering (S) Mean ± SD (95% CI)	P-value (Student`s t-test)
Flexural Strength (Mpa)	(K) Katana ™ Zirconia	(Non-Aging)	2162± 406.0 (1736; 2588)	2130± 456.5 (1651; 2609)	0.8983 (NS)
		A (Aging)	2111± 118.4 (1987; 2235)	2168± 212.4 (1945; 2391)	0.5808 (NS)
		P-value (Paired t-test)	0.7782 (NS)	0.8822 (NS)	

NS; Insignificant Different at P≤ 0.05

Table (4): Average mean grain size (nm)

Average grain size(nm)		Normal (N) Sintering Mean ± SD	Speed (S) Sintering Mean ± SD
Katana utml	(Non-Aging)	2.6 ±0.56 nm	1.8 ±0.50 nm
	(Aging)	2.8 ±0.69 nm	2.4 ±0.49 nm

Scanning electron microscope are presented in figures 1-4.

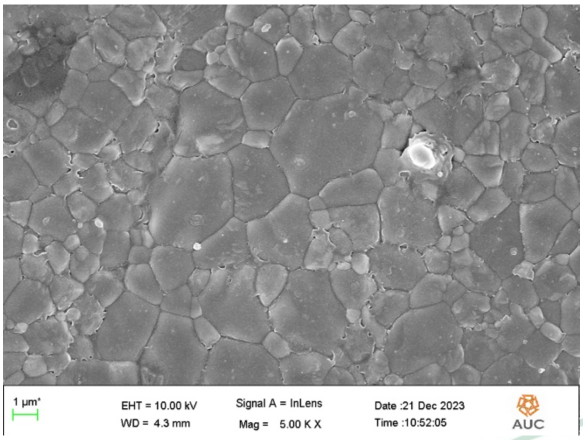

Fig 1: Katana UTML Normal cycle

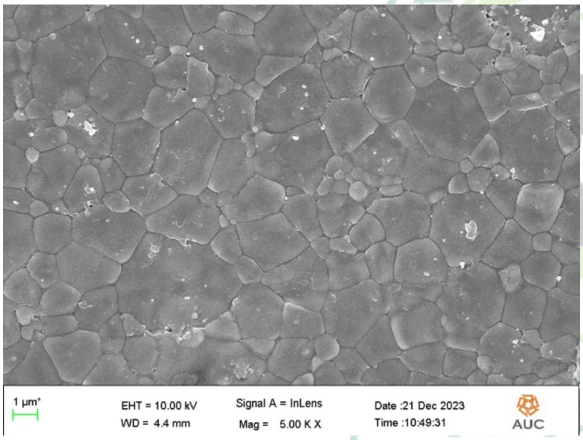

Fig 2: Katana UTML Speed cycle

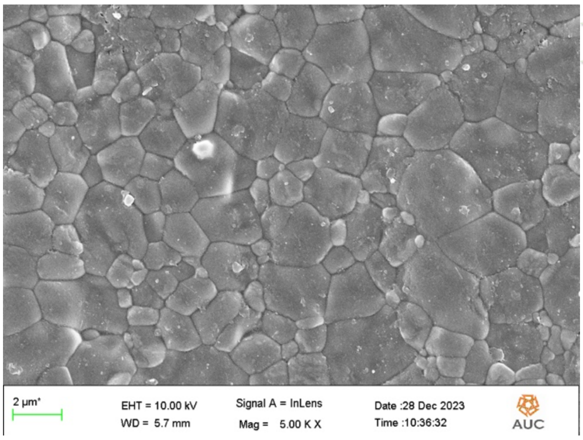

Fig 3: Katana UTML Normal Cycle, Aged

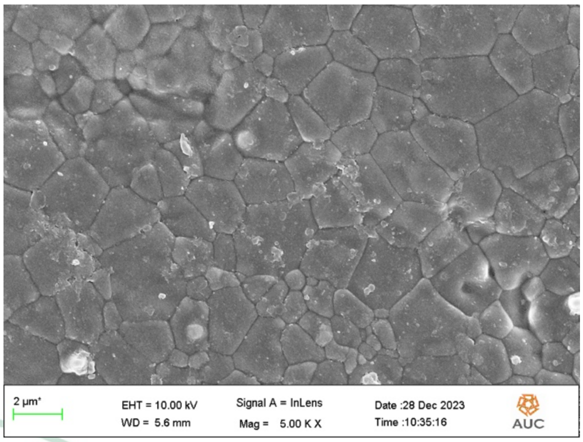

Fig 4: Katana utml speed cycle, aged

Discussion

Then Influence of rapid sintering cycles and accelerated aging on biaxial flexural strength (BFS) of Katana UTML cubic zirconia has been reported in this study. Results reported that both rapid sintering protocols and accelerated aging did not show to have a negative impact on the biaxial flexural strength of Katana UTML cubic zirconia. while Average mean grain size values showed a reduction in size after subjecting to speed sintering protocol. Hence, the null hypothesis was partially rejected.

Mechanical and optical properties of zirconia greatly depend on its microstructure. It is worth mentioning that sintering parameters such as time and temperature have a direct effect on the grain growth, where elongated time and elevated ranges of temperatures may result in larger grain size. However, larger grain size may not affect negatively on the mechanical properties till a certain level. In other words, according to zirconia formulations or microstructure a suitable sintering parameter should be applied to secure a restoration with desired density and strength. As using temperatures below or above the desired range may result in incomplete grain growth or even larger grain to a degree that will affect negatively on mechanical properties. [21]

The results of this study were in agreement with a previous study conducted by Ebeid et al [28] that applied different sintering parameters and reported a non-significant difference in biaxial flexural strength values of translucent zirconia (Bruxzir, 3Y- TZP). The same results were also reported by Hjerppe et al [29] who used a different temperature during rising and holding stages for yttrium partially stabilized zirconia (ICE Zirkon, Y-TZP) and concluded that altering the sintering parameters did not affect negatively on the biaxial flexural strength values. While Several studies [26,28-31] were in agreement with this study findings, it worth noting that the effect of speed sintering protocols or altering the sintering parameters may vary according to different formulations used during zirconia fabrication.

Stawarczyk et al [21] in a previous study reported a reduction in strength with 3Y- TZP that was sintered above 1550 °C, and proposed that elevated sintering temperature resulted in opening or pores between the zirconia grains led to increase porosity and affected negatively in zirconia density, as a result reduction in mechanical properties was obtained. It is crucial to point out that speed sintering cycles uses an elevated ranges of temperatures and higher rates for heating and cooling, that could result in porosities at grain boundaries as a result of incomplete grain development or growth, in addition to intergrain porosities that may be obtained from insufficient fusion or union of small grains.

In agreement with this study Cokic et al [25] evaluated the effect of rapid sintering cycles on both (CEREC Zr / 3Y-TZP, InCoris TZICS) and (Kantana STML/5Y-PSZ) zirconia. Regarding (Kantana STML/5Y-PSZ) speed sintering cycles showed similar or comparable biaxial flexural strength values, in addition to a similar density and microstructure, and proposed an explanation as the amount of Y_2O_3 and its tetragonality remains the same after speed sintering cycles in addition to, speed sintering cycles did not reveal to have any impact on the phase composition of cubic 5Y-PSZ. Thus, the influence of speed sintering on the mechanical properties of cubic zirconia was not significant.

The results of the current study did not align with previous studies [25-27] suggesting that speed sintering programmers may be critical and should be used in specific and recommended furnaces, as high rates for heating and cooling applied may provoke a thermal shock causing high tensile stress at surface that may overcome the strength of the ceramic leading to microcracks causing a materials failure. Moreover, rapid or accelerated heating may lead to the formation of what is known as a core-shell formulation or structure characterized by a dense surface with remarked porosity inside the material, resulting in decreased mechanical and optical properties as well. That is why speed or rapid sintering cycles should be applied with specific recommendations regarding furnaces and restorations designs.

The effect of hydrothermal aging on biaxial flexural strength of Katana UTML cubic zirconia was evaluated in this study. Accelerated aging in this study was performed for 5 hours in an autoclave to simulate about 15 years of intraoral or clinical application. The results reported that hydrothermal aging did not show to have a negative effect on the biaxial strength values.

Monolithic restorations fabricated in a full anatomical state without the need for veneering materials, so its subjected directly to intraoral environment that may triggers low temperature degradation. In addition to, monolithic restoration showed to have a reduced alumina content to enhance translucencies where alumina rule in resisting aging is crucial. As a result, monolithic zirconia may be affected easily by low temperature degradation (LTD). It is worth mentioning that the effect of LTD on tetragonal zirconia is clear, where a spontaneous tetragonal to monoclinic phase transformation occurred with remarked increase in monoclinic phase percentage after aging was reported. Results in a decreased mechanical property due to the pulling out of grains and micro cracking.

The results were in agreement with Yan M et al [35], where the microstructure, grain size and flexural strength values for two cubic zirconia materials /5Y-PSZ did not show a significant difference between normal and rapid sintering even after accelerated aging using an autoclave for 5 hours / 134°C and a water pressure with 0.2 MPa. This Could be explained as 5-mol% (5Y-TZP) accommodates a higher fraction of the cubic phase, that may not be affected regardless the applied sintering protocol [37], moreover the cubic phase confers better resistance to low temperature degradations and did not undergo a tetragonal to monoclinic phase transformation unlike 3Y-TZP. [30] Brian et al [38] concluded that hydrothermal aging of yttria-stabilized tetragonal zirconia showed a significant difference after prolonged aged time for 50,100,200 hours that considered to be clinical irrelevant, and till 5 hours the effect was not significant. This result was in agreement with the results of this study.

Scanning electron microscope inspection in this study showed a reduced average grain size after using a speed cycle protocol. In agreement with the SEM finding of this study Attia et al [39] reported a reduction in mean grain size of after subjecting the samples to rapid sintering cycles.in addition it was reported previously that speed sintering could result in a more uniform with reduced grain size for different zirconia formulations. [23] Moreover, the microstructure of the discs subjected to aging did not report a significant difference, this could be explained as cubic zirconia can resist low temperature degradation that was also supported by the results that did not show a significant difference in biaxial flexural strength after subjecting to hydrothermal aging.

Scanning electron microscope analysis also reported a consistent microstructure within the blank thickness that was also reported and confirmed with the biaxial flexural strength values that showed a comparable result between the sectioned discs. So, the variation between the four different layers that provides a graduation in chroma within the blank depends on color pigments rather than different zirconia formulations.

The primary concern about speed sintering protocols is the potential for insufficient time to allow optimal grain growth leading to a material with inadequate densification, while conventional sintering protocols consumes more time. The results of this study showed no significant difference between both sintering cycles may be due to that the sintering protocol was performed according to the manufacture recommendations and discs were sintered in a speed sintering furnace. This could be considered as a limitation for this study.

Conclusions

1. Speed sintering protocols for katana UTML cubic zirconia and hydrothermal aging did not show to have a negative impact on the biaxial flexural strength.
2. Speed sintering protocols reported a reduction in average grain size for katana UTML.

Recommendation

Different sintering protocols rather than the recommended cycles may be applied, longer aging periods with different aging conditions may also be recommended.

Acknowledgment

Sincere gratitude to Dr. shady M. EL Naggar, Associate professor of Removable Prosthodontics at Badr University in Cairo, for his contribution in providing statistical methodology of the research.

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