

## Effect of Variable Holding Time on Biaxial Strength of Conventional Sintering 4Y and 5Y PSZ

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### ABSTRACT

**Background:** The biaxial strength of dental zirconia restorations is strongly influenced by sintering environments. Variations in sintering protocols could affect zirconia materials' flexural strength.

**Aim of the study:** To examine the effects of varying sintering holding time settings on 4Y and 5Y PSZ translucent zirconia's biaxial flexural strength.

**Materials & Methods:** A digitally designed 3D STL file of disc sample was made, then using CAD CAM, the disc specimens were milled out of 4Y PSZ super translucent multilayered STML and 5Y PSZ ultratranslucent multilayered UTML zirconia ceramics (Kurary Noritake). Specimens of both two main ceramic groups were distributed into 3 subgroups and subjected to 1, 2 and 3 hours holding sintering time at 1550°C. After sintering, samples were subjected to piston on 3-balls biaxial flexural strength tests, load was applied until failure occurred, strength results were statistically analyzed utilizing SPSS program and the findings were compared among groups through the use of 1-way ANOVA and T tests, level of significance was set at 0.05.

**Results:** According to our study findings, the 4YSTML 2 hours holding revealed highest flexural strength for tested zirconia disc samples, whereas the lowest strength values was for 5YUTML 1 hour holding.

**Conclusions:** At 1550°C sintering temperature, translucent 4Y and 5Y PSZ zirconia showed highest biaxial flexural strength when sintered conventionally for 2 hours, the strength was affected negatively by increased and decreased holding time.

**KEYWORDS:** Holding time, Sintering, Strength, Translucent zirconia, Yttria.

### INTRODUCTION

As a result of rising aesthetic demands and technological advancements, the needs for high strength are biologically acceptable alternatives to porcelain fused to metal restorations having have been grown [1]. Zirconia ceramic restorations offers superior cosmetic performance and reduces the possibility of metal allergies problems as compared to traditional ceramic fused to metal restorations [2]. Furthermore,

restorations fabricated from zirconia having better mechanical qualities compared to other all ceramic restorations such lithium disilicate and hybrid ceramics [3].

Clinicians found zirconia ceramics to be the most appealing dental ceramic due to their exceptional mechanical and esthetic characteristics. The first introduced zirconia was 3 mol% yttria tetragonal zirconia polycrystals (3Y TZP) which exhibits the greatest strength (more than 1,100 MPa) of all dental zirconia, high strength occurs as a result of the toughening due to phase transition of zirconia from monoclinic (m) to (t) tetragonal phase. Unfortunately, 3Y TZP's translucency is not quite as high as that of a human tooth, therefore an esthetic porcelain veneering material always needed to improve esthetic [4, 5].

Actually, low translucency was the most distinctive factor that affects esthetic of earliest 3Y TZP zirconia. Fortunately, the dental manufacturers succeeded to boost zirconia translucency by incorporating the cubic phase (c phase) and increasing yttria content, therefore 4Y and 5Y PSZ (4 and 5 yttria partially stabilized zirconia) were introduced, but unluckily they have lower strength than earlier 3Y TZP [6].

Zirconia mechanical characteristics enhanced due to the strengthening phenomena of conversion through a phase transformation mechanism was heat induced [7]. Typically, zirconia sintered using an indirect heating method that uses resistive heater components to heat the air inside the furnace, which in turn increases the temperature of the zirconia. Because materials based on zirconia have a low degree of thermal conductivity, heat moves slowly from the surface to the inside of the restoration [8]. Due to the fact that quick heating and cooling might causes cracking [9, 10], a slow rate of heating and cooling (8°C/min) accompanied with a prolonged holding time of two hours must be utilized for consistent heat distribution and ideal sintering [11].

Whereas zirconia's longevity has been the subject of recent research's examining the material's mechanical characteristics [12]. The focusing on the significance of heat effect on zirconia strength was grown in the field of dentistry, as previous researchs produced a conflicting result, some researchs observed that the flexural strength was not influenced by alterations of sintering settings [13]. On the other hand, other studies showed that variations in zirconia's sintering settings have a direct effect on its specific characteristics and microstructure [1]. Increased flexural strength of the esthetic zirconia could result from extending the holding period of sintering, according to Juntavee et al. [14]. While Ebeid et al. [15] found that variations in the sintering periods have no effect on biaxial flexural strength. Moreover, in order to improve the flexural strength of zirconia, Ersoy et al. [16] suggested combining a higher temperature with a brief sintering period.

Only a few researchs examined the mechanical behavior of high yttria content translucent zirconia sintered conventionally at variable holding periods, although basically the translucent 4Y and 5Y PSZ zirconia are the most widely used esthetic materials and vast majority of monolithic zirconia restorations were made from these two brands due to ceramic chipping issues relate to 3Y TZP, in addition to fact that they have a lower strength than 3Y TZP, so any sintering variables decreases their strength is detrimental and fracture possibility of restoration increased highly. The purpose of this work was to study the influence of three varying holding times conventional sintering on the biaxial flexural strength of two different yttria content dental zirconia. The null hypothesis stated that strength of zirconia restorations will not affected by varying sintering times parameter.

## MATERIALS AND METHODS

### Sample preparation

Utilizing CAD CAM technology, a standard tessellation language (STL) file for disc sample was designed for specific geometry 12 mm diameter and 1.2 mm thickness, then the files were sent to the milling machine (Coritec 250i, Imes-Core, Germany). A total ninety-disc shape samples were made, specimens were divided into two main groups (n =45). Super translucent multilayered zirconia STML blocks 4Y% yttria were used to produce first main group, while the other main group samples were fabricated from ultratranslucent multilayered zirconia UTML block 5Y% yttria. The forty-five STML samples then subdivided into 3 subgroups (no=15) relies on sintering time (1, 2 and 3 hours) and UTML specimens also divided in the same manner. All zirconia samples were made from same manufacturer (Katana Zirconia; Kuraray Noritake Dental Incorporation, Tokyo Japan).

After milling, the surfaces of disc specimens were dry polished with 1200 grit silicon carbide abrasive (SiC abrasive paper P1200; Buehler USA). Sintering was performed using Vita Zircomat 6000 MS zirconia sintering furnace (Vita Zahnfabrik, Germany). Heat increase and cooling rates of sintering were 8°C per minute and the final sintering temperature was 1550°C as recommended from manufacturer [17], the control panel of the furnace was used to modify the sintering time. The sintered groups and sintering protocols were summarized in Table 1.

After sintering, thickness and diameter dimensions of samples were checked using a digital caliper micrometer (Digimatic, Mitutoyo, Japan), then samples were ultrasonically washed with distilled water and isopropyl alcohol for five minutes.

**Table 1** (Group's classification of zirconia disc specimens depending on holding time.)

| Group    | Heating rate<br>degrees in<br>minute | Final sintering<br>temperature in<br>degrees | Sintering<br>time in<br>hours | Cooling rate<br>degrees in<br>minute |
|----------|--------------------------------------|----------------------------------------------|-------------------------------|--------------------------------------|
| 4YSTML 1 | 8                                    | 1550 <sup>0</sup>                            | 1                             | 8                                    |
| 4YSTML 2 | 8                                    | 1550 <sup>0</sup>                            | 2                             | 8                                    |
| 4YSTML 3 | 8                                    | 1550 <sup>0</sup>                            | 3                             | 8                                    |
| 5YUTML 1 | 8                                    | 1550 <sup>0</sup>                            | 1                             | 8                                    |
| 5YUTML 2 | 8                                    | 1550 <sup>0</sup>                            | 2                             | 8                                    |
| 5YUTML 3 | 8                                    | 1550 <sup>0</sup>                            | 3                             | 8                                    |

### Strength test

Biaxial flexural tests accomplished by placing the disc samples on 3 steel balls and 1.2 mm diameter piston applies load on the center of the specimen at a crosshead speed of 0.5 mm/minute until fracture occur (Figure1) for both STML and UTML groups as specified by (ISO) standard 6872 [18]. A universal testing machine (Zwick Z010, Ulm, Germany) was used to perform the test and the highest load at fracture of samples was established using standard formula listed below [19, 20].

$$S = -0.2387P(X-Y)/d^2$$

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

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

**S**= Biaxial flexural strength in (MPa).

**d**= Sample thickness.

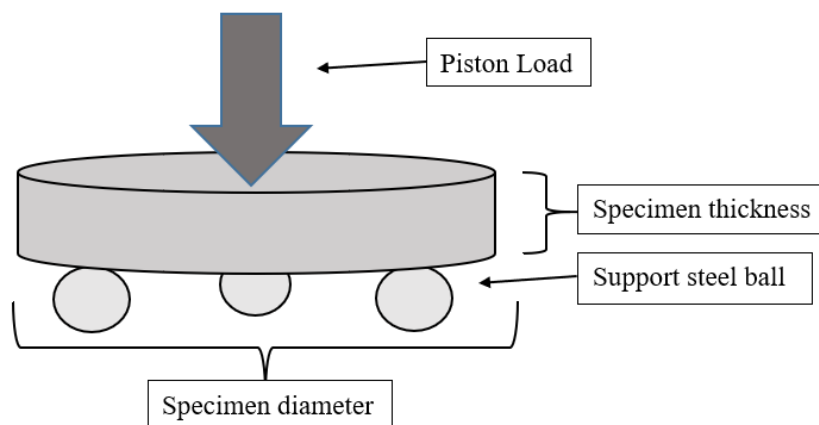
**v**= Poisson ratio of ceramic 0.238

**P**= load at fracture in newton.

**r<sub>1</sub>**= Radius of support circle.

**r<sub>2</sub>**= Radius of loaded area.

**r<sub>3</sub>**= Radius of sample.



**Figure 1.** (Schematic illustrates piston on three balls test for biaxial strength of zirconia disc specimen of 12 mm diameter and 1.2 mm thickness.)

## Statistical analysis

The statistical analysis was conducted using SPSS program (SPSS IBM Corp., USA). Mean values and standard deviations of data collected for test groups have been listed. One-way ANOVA and Tukey test were employed to compare differences in flexural strength among groups and the level of statistical significance was set at 0.05.

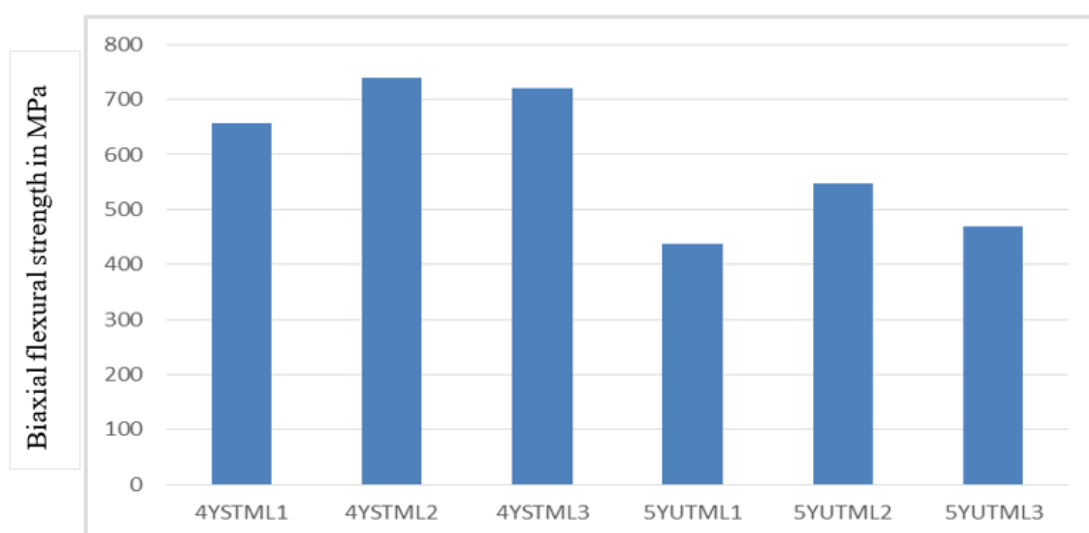
## RESULTS

Mean biaxial strength values and standard deviations (SD) for tested groups are listed in Table 2. Regarding biaxial strength, one-way ANOVA showed significant differences among groups sintered at variable holding times ( $P \leq 0.05$ ). Tukey's tests revealed that there was a significant difference in the strength as the sintering time varied, and the strength was decreased when holding time was shorter and longer than 2 hours within each main group. Highest results of our study was for 4YSTML2 hours subgroup while the lowest strength results was for 5YUTML1 hour subgroup. Mean strength of STML three subgroups was higher than means of all UTML subgroups (Figure 2). Each subgroup sintered at 1 hour holding time revealed a lowest strength than others in their main group, in addition to that subgroup sintered at 2 hours holding showed highest strength results among others in their main group.

**Table 2** (Mean biaxial flexural strength in MPa and standard deviation SD mechanical test results of groups.)

| GROUP    | Biaxial Flexural Strength Mean (MPa) | SD   |
|----------|--------------------------------------|------|
| 4YSTML 1 | 657.46*                              | 6.65 |
| 4YSTML 2 | 739.00 *                             | 5.34 |
| 4YSTML 3 | 719.40*                              | 6.15 |
| 5YUTML 1 | 438.33*                              | 7.28 |
| 5YUTML 2 | 547.13*                              | 7.11 |
| 5YUTML 3 | 469.33*                              | 5.49 |

\*Statistically significant,  $P < .05$



**Figure 2** (Mean biaxial strength among tested groups of disc zirconia samples.)

## DISCUSSION

Monolithic restorations made from 4Y and 5Y PSZ have a higher yttria content and more translucent than others fabricated from 3Y TZP, this type of zirconia ceramic have been released to the market for improving aesthetics, manufacturing simplicity and productivity [21].

An effort has been made recently for dental restorations to be produced chairside utilizing speed sintering protocols, which usually involves quick heating and rapid cooling with short holding period at the appropriate sintering temperature enabling short total sintering process [22].

Distinctly, speed sintering eliminates the necessity of temporaries, facilitates the making restorations in the clinic at single visit, as they do not need to schedule a second appointment to receive the restoration, so it saves both dentists and patient time. Furthermore, it requires less energy consumption and shorter manufacturing time compared to conventional sintering protocols [15]. Nevertheless, it is still uncertain if speed sintered PSZ is appropriate for clinical usage because its mechanical and microstructural characteristics might vary from PSZ which conventionally sintered [16].

According to the manufacturer's recommendations, PSZ to reach its maximum density must be sintered conventionally at slow heating and cooling cycles with long holding period at certain temperatures 2 hours and 1500<sup>0</sup>–1550<sup>0</sup> degrees Celsius, respectively, in order to acquire the best mechanical properties of material [23, 24].

The purpose of this in vitro study was to reveal possible influence of changing sintering time on strength of conventionally sintered high yttria content zirconia specimens. The fundamental motive for conducting this research was that different yttria content zirconia can behave differently regarding strength characteristics when the sintering duration is adjusted, achieving a higher strength material benefits clinically of restorations keep working inside the patient's mouth for a longer period of time.

Biaxial flexural strength test is the one of the mechanical characteristics utilized to assess the clinical efficacy of zirconia dental materials over an extended period of time [25]. A piston on 3-balls tests was carried out to determine the biaxial flexural strength (BFS). Since this technique previously used for a long period, several researchs attempted to adapt this test with regards to the sample dimensions and shape [26, 27].

Based on the BFS results, a statistically significant differences were discovered between the groups in the current investigation, since sintering duration had an impact on flexural strength, so the null hypothesis stating that varying sintering times would not have an effect on zirconia strength was rejected.

The data reveals that the flexural strength levels of 4Y and 5Y PSZ were less than 1000 MPa which comparable to those from previous investigations [6, 28], as these studies reports that high yttria content and more cubic phase 5Y PSZ material has the biaxial flexural strength less than 600MPa and 800MPa for 4Y PSZ material, these materials were offered to be most appropriate for full anatomic zirconia restorations in the anterior region and sometimes may be used as single unit restoration in posterior area [17]. They contained more cubic phase, and less tetragonal transformational toughening compared to earlier 3Y TZP so their mechanical characteristics have changed dramatically [6, 29]. Lower fracture resistance and strength reduction of the newer more translucent zirconia materials which containing more cubic phase may have a greater significance in clinician's decisions to use them in scenarios where high strength zirconia used traditionally.

Manufacturing company states that 4YSTML has higher strength than 5YUTML [17], this consistent with results of our study which revealed that 5YUTML three subgroups have a lower strength of than all 4YSTML subgroups, the explanation of this relied on facts that 5Y incorporates higher yttria content and includes more low strength cubic phase, so a lower tetragonal phase transformation toughening that leads to a decrease in BFS [30].

The 4YSTML 2 hours holding period subgroup has the highest flexural strength values for all subgroups tested, this may be related to that 4Y zirconia has more tetragonal and less cubic phases than 5Y,

alongside this group sintered as recommended from manufacturer regarding both holding time and temperature, consequently, not a surprise that 4YSTML 2 hours tested group achieved highest strength than other 4YSTML subgroups and the all 5YUTML subgroups. These results were confirmed by Kulyk et al study which found that at 2 hours holding the sintered zirconia attain highest strength due to greatest amount of tetragonal gained which improves material strength [31].

The two subgroups sintered at 3 hours showed less strength than 2 hours holding subgroups. The detrimental effect of increasing holding time results from our study agrees with other study [32] showed that extended sintering time may result in more microscopic pores, which could weaken zirconia's and reduce material strength. However, longer period sintering at higher temperatures yield more energy affect the grain size and cause uneven distribution of yttria [33], this undesirable effect has a negative impact on flexural strength especially for higher yttria content zirconia [30].

Lowest strength of tested specimens was for 5YUTML 1 hour subgroup, this most likely due to fact that 5Y zirconia have more cubic phase so a reduced percentage of tetragonal phase, furthermore a less sintering time contributes less phase transformational toughening causing synergistic effect in reduction of samples strength. This confirmed by another study [34] revealed that 5Y zirconia has lower strength when sintered for short time compared to long holding period, a longer holding time favors sintering to seal gaps and void present in the white-body and flaws which present at the edges, and this may be the possible explanation why 5YUTML 3 hours has higher strength than 5Y UTML 1 hour holding.

Both subgroups sintered at 1 hour holding showed lowest strength values than others in their main group this associated to less sintering time, these results coincide with other studies demonstrated that steady heat and cool rates with adequate holding time at final temperature are essential for acquiring most appropriate zirconia density and microstructure [35,36]. Insufficient sintering can cause a lot of porosities and huge pores inside the sintered zirconia, which could play a crucial rule in reducing material strength [37]. This may explains why 1 hour holding time produces weaker zirconia as its strength strongly relies on transformation toughening mechanism which is strictly associated to the grain size and content of tetragonal phase [38].

Our results not agree with Hjerppe et al. study [39], which found that strength not significantly affected when different holding times were used for sintering zirconia, this possibly clarified as varying yttria content of zirconia brand used and sintering temperature used, as latter study used 3Y TZP discs from different manufacturer (ICE Zirkon, ZirkonZahn, Italy), in addition to that samples were subjected to a different sintering temperature 1500°C which lower than temperature which employed in our study, Kulyk et al [31], found that higher yttria content zirconia has a different manner of response to heat compared to 3Y TZP and strength decreases as sintering temperature increase above 1500°C, while for the 3Y zirconia ceramic, the strength is almost linearly related where strength increases when temperature increase from 1500°C to 1550°C if both were sintered for 2 hours [40].

The limitation of this study was that zirconia blocks which have been used in our investigation were brands from same company, as the results possibly not relevant for other brands from different manufacturers which may have different grain sizes and requires a different sintering specification. The sintering speed and temperature were also might affects the strength of the zirconia material and the effect these parameters were not studied. Additionally, the mechanical test used in this study was static load but in reality the cyclic loading tests are more closely representative to masticatory forces inside oral cavity so further dynamic fatigue tests are required.

## CONCLUSION

### Following findings were drawn considering the results of this study:

1. At 1550°C, the biaxial flexural strength of conventionally sintered 4Y% PSZ and 5Y% PSZ ceramics is a characteristic sensitive to sintering time changes, whereas decreasing is more detrimental than increasing holding time.
2. Highest flexural level of strength was reached when holding time was 2 hours, more or less than this time the strength will be decreased.
3. For high yttria content zirconia the effect of short sintering duration is more harmful than increasing holding time regarding biaxial flexural strength.
4. Higher yttria content 5Y% zirconia more affected than 4Y% zirconia regarding material strength when sintering time decreased.

### Conflict of interest

The author declare that they have no conflict of interest with this work.

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