

THE EFFECT OF DIFFERENT SINTERING CYCLES ON THE TRANSLUCENCY AND BIAxIAL FLEXURAL STRENGTH OF MONOLITHIC ZIRCONIA (IN VITRO STUDY)

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ABSTRACT

INTRODUCTION: In order to make zirconia a chair-side option, it was essential to reduce the sintering time without altering the material's physico-mechanical properties.

The aim of the study: This study aims to evaluate the effect of speed and super-speed sintering compared to conventional sintering on monolithic zirconia, regarding the translucency and biaxial flexure strength.

MATERIALS AND METHODS: Forty-five 15 mm x 1.5 mm Y-TZP zirconia discs were fabricated and divided into three groups (n=15) according to sintering time; group SS: super-speed sintering, group S: speed sintering and group CV: conventional long-term sintering. The specimens were tested for any changes in translucency, then subjected to a biaxial flexure strength test.

RESULTS: Conventional, speed and superspeed sintering revealed no significant difference in the biaxial flexure strength of the zirconia discs. However, super-speed sintering produced the least translucent specimen followed by conventional sintering, and speed sintering gave the best results.

CONCLUSION: Zirconia can be used as a chairside material using the superspeed sintering protocols for restoration in the non-aesthetic zone. Longer sintering protocols are essential to obtain highly translucent restorations.

KEYWORDS: monolithic zirconia, Y-TZP zirconia, speed sintering, super-speed sintering, biaxial flexure strength, translucency

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INTRODUCTION

Good aesthetics, biocompatibility and excellent mechanical properties are some of the reasons why Yttrium-stabilized tetragonal zirconia polycrystals (Y-TZP) is becoming the material of choice for fixed restorations.^[1, 2]

When first introduced, Zirconia's main disadvantage was its high opacity, therefore it was used as a core material, veneered with feldspathic porcelain,^[3] resulting in cohesive failure caused by residual stresses and causing chipping in the veneer layer,^[4] hence, the necessity to develop translucent monolithic zirconia restoration material.^[5-8]

First to introduce a zirconia-based material to the dental market was Vita Zanhfabrick, Germany as the in-ceram zirconia which had improved structural strength by 30-40% due to its zirconia content. Soon after, pre-sintered ceramic blocks were milled using CAD/CAM milling devices.^[9]

The main properties needed for monolithic zirconia restorations are high strength, density, and translucency. These properties are affected by the

sintering conditions since the degree of densification of the ceramic powder strongly affects the physico-mechanical properties of zirconia. This densification occurs while sintering the material from compressed powders at high temperatures, below their melting point.^[10]

The conventional fabrication of Y-TZP restoration is a tedious process; milling is performed in one day, sintering is done over-night, as it requires very slow heating and cooling (5-10°C/min) along with a dwell time of several hours, and the final processing is left to the next day.

However, in order to meet the demand of a time-effective chair-side CAD/CAM fabricated restorations, new sintering furnaces that can accommodate shorter sintering cycles have been developed. These short-time or speed sintering cycles embrace new prospects as a restoration can be milled and sintered in less than a day.^[11, 12]

This study will be an attempt to evaluate the effect of speed, super-speed, and conventional sintering on the properties of monolithic zirconia.

The null hypothesis of this study is that there would be no difference between the study groups after the different sintering techniques used regarding the biaxial flexural strength and translucency.

MATERIALS AND METHODS

In this study, inCoris TZI translucent zirconium oxide (Sirona Dental Systems Inc, NY, USA) and IPS e-max Ceramic glaze powder and stain (Ivoclar Vivadent, Schaan, Liechtenstein) were used. The composition of each of the previous materials is mentioned in Table 1.

A total of 45 zirconia disc specimens of 15mm in diameter and 1.5mm in thickness were cut using a diamond disc mounted on a microtome (IsoMet 4000, buehler, Lake Bluff, IL, USA). The pre-sintered zirconia discs were randomly allocated into three equal groups according to the sintering technique used (n=15);

Group (S): Zirconia discs were sintered using the speed sintering cycle in a Sirona Inlab Profire Speed Sintering furnace (Sirona Dental Systems Inc, NY, USA) according to the manufacturer instructions.

Group (SS): Zirconia discs were sintered using super-speed sintering cycle in a CEREC SpeedFire sintering furnace (Sirona Dental Systems Inc, NY, USA) according to the manufacturer instructions.

Group (CV): This is the control group where zirconia discs were sintered using conventional sintering cycle in a Sirona Inlab Profire Speed Sintering furnace (Sirona Dental Systems Inc, NY, USA) according to the manufacturer instructions.

All sintered zirconia discs were glazed using the IPS-emax glaze. Each disc was then tested for translucency and biaxial flexure strength.

Translucency

The specimens were evaluated for their translucency by obtaining the translucency parameter. Vita easyshade V Spectrophotometer (VITA Zahnfabrik, H. Rauter GmbH & Co. KG Bad Säckingen, Germany) was used to measure the L, a, & b values for each specimen against a white and a black background. These values were used to calculate the translucency parameter for each specimen, using the following equation:^[13]

$$TP \text{ value} = \{(LW - LB)^2 + (aW - aB)^2 + (bW - bB)^2\}^{1/2}$$

Where W=white background, B=black background and L, a & b represent the colour space of the CIE system defined by three values. The CIE L* value is a measure of the lightness of an object, the CIE a* value is a measure of redness or greenness (positive or negative value), while the CIE b* value is a measure of yellowness or blueness (positive or negative value).

A high TP value indicates high translucency and low opacity^[14].

Biaxial Flexural Strength

The biaxial flexure test was employed according to ISO 6872.

The sample holder for the biaxial flexural strength test comprised three tempered steel balls with a diameter of 3.2 mm. The steel balls formed an equilateral triangle with an edge length of 10 mm and the ball support circle was 120°. The centre of the specimens, which were put upon the steel balls and the centre of the equilateral triangle were aligned coaxially. After the positioning the specimen's centre was loaded from above with a plunger with a diameter of 1.2 mm until failure, using a universal testing machine (by AGS-X 5 KN, shimadzu, Japan) with 0.5mm/min crosshead speed^[15, 16].

The biaxial flexural strength was calculated as:

$$\sigma = \frac{-0.2387P(X - Y)}{d^2}$$

Where σ : biaxial flexural strength (MPa); P: fracture load (N); d: specimen disc thickness at fracture origin (mm).

And X and Y are calculated as follows:

$$X = (1 + \gamma) \ln \left(\frac{B}{C} \right) + \left[\frac{(1 - \gamma)}{2} \right] \left(\frac{B}{C} \right)^2$$

$$Y = 1 + \gamma \left[1 + \ln \left(\frac{A}{C} \right)^2 \right] + (1 - \gamma) \left(\frac{A}{C} \right)^2$$

Where γ : Poisson's coefficient (ceramic = 0.25, ISO 6872); A: radius of support circle (mm); B: radius of loaded area (mm); C: radius of specimen disc (mm).

RESULTS

The results obtained in this study regarding the translucency parameter (TP) and the biaxial flexure strength (σ) for yttrium stabilized monolithic zirconia after sintering using three different types of furnaces: super speed sintering, speed sintering and the conventional sintering furnace, were found to be as follows:

1. Translucency test

TP values were calculated using the equation mentioned previously for the specimens after the sintering process. Figure 1 shows the individual TP values for each specimen.

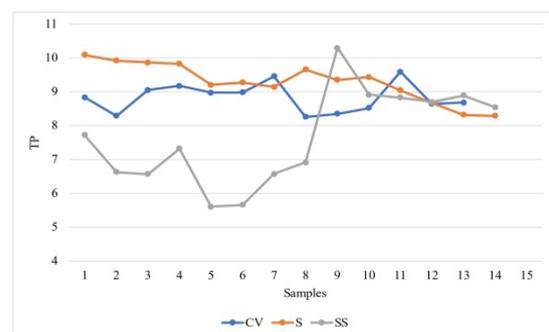


Figure 1: TP values for each individual sample in the three groups after sintering. TP values calculated according to Error! Reference source not found.

Table 2 shows the means, variance, and standard error for the three sintering techniques. Single-factor one-way ANOVA test ($\alpha = 0.05$) concluded that there was a significant difference between the mean values ($p = 0.000101$). Table 3 shows the pairwise t-tests' p values. All p values for the t-test were smaller than the α -value, thus ensuring the statistically significant difference between the groups.

2. Biaxial flexure strength test

The biaxial flexure strength test was done according to ISO 6872 and the maximum load for each specimen was recorded as shown in Figure 2. Single factor one-way ANOVA test was performed on maximum load values, and it concluded that there was no significant difference between the maximum load endured by the discs after sintering in the three different furnaces ($p=0.971$).

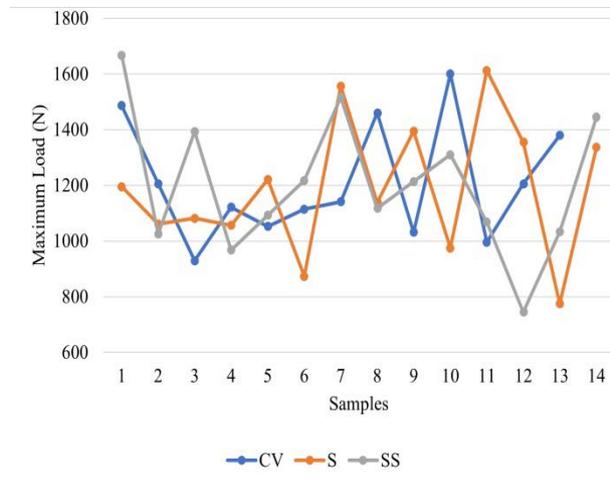


Figure 2: Maximum load for each specimen

Table 2 shows the means, variance, and standard error of the maximum load for the three sintering techniques. Table 3 shows the pairwise t-tests' p values. All p values for the t-test were higher than the α -value, thus ensuring the statistical non-significance between the groups. The biaxial flexure strength (σ) values were calculated from the maximum load values for all specimens after the different sintering processes. Figure 3 shows the individual σ values for each specimen.

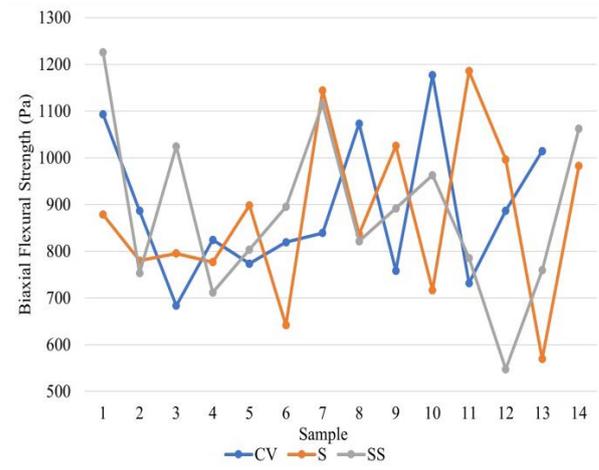


Figure 3: The individual σ values for each specimen.

Table 2 shows the means, variance, and standard error of the biaxial flexure strength for the three sintering techniques. Single-factor one-way ANOVA test ($\alpha = 0.05$) concluded that there was no significant difference between the mean values ($p = 0.971$). Table 3 shows the pairwise t-tests' p values. All p values for the t-test were higher than the α -value, thus confirming the statistical non-significance between the groups.

Table 1: Composition of materials used in this study

Material	Manufacturer	Composition
InCoris TZI blocks	Sirona Dental Systems Inc, NY, USA.	ZrO ₂ +HfO ₂ +Y ₂ O ₃ ≥ 99.0% Y ₂ O ₃ > 4.5 - ≤ 6.0% HfO ₂ ≤ 5% Al ₂ O ₃ ≤ 0.5% Other oxides ≤ 0.5%
IPS e.max® Ceram glaze	Ivoclar Vivadent, Liechtenstein	SiO ₂ , Li ₂ O, Na ₂ O, K ₂ O, Al ₂ O ₃ , CaO, P ₂ O ₅ , F

Table 2: showing statistical information for the TP test, the maximum load, and the biaxial flexure.

Tested Parameter	Group	Mean	Variance	Std Err	Minimum	Maximum
TP test	C	8.831	0.182	0.255	8.316	9.346
	V	9.292	0.325	0.245	8.795	9.788
	SS	7.653	1.968	0.245	7.156	8.149
Maximum load	C	1209.570	43608.830	64.860	1078.270	1340.880
	V	1187.990	59396.830	62.500	1061.460	1314.520
	SS	1200.840	60216.880	62.500	1074.310	1327.370
Biaxial flexure strength	C	889.340	23574.930	47.690	792.800	985.890
	V	873.470	32109.930	45.960	780.440	966.510
	SS	882.930	32553.250	45.960	789.890	975.960

Table 3: The pairwise t-tests' p values.

Tested Parameter	group 1	group 2	p-value
TP test	CV	S	0.0252
	CV	SS	0.0088
	S	SS	0.0008
Maximum load	CV	S	0.8065
	CV	SS	0.9213
	S	SS	0.8905
Biaxial flexure strength	CV	S	0.8065
	CV	SS	0.9213
	S	SS	0.8905

DISCUSSION

The aim of this study was to evaluate the effect of sintering speed on the translucency and biaxial flexure strength of Y-TZP, to provide a framework for the new sintering cycles, allowing dentists nowadays to deliver chairside zirconia restorations. Zirconia restorations used to be Y-TZP core veneered by feldspathic porcelain to achieve the desired aesthetics requirements of the restorations, such as colour and translucency. The major concern was then the chipping of the veneering ceramic. Thus monolithic zirconia systems, including a single material without additional veneering were developed to be used by CAD/CAM technologies to overcome these drawbacks.^[17]

The use of monolithic zirconia used at the present time in the dental field, has higher mechanical and optical properties, allowing clinicians to perform more conservative preparations compared to conventionally veneered ceramic or metal-ceramic restorations^[18]

However, the prolonged sintering time of the conventional sintering furnaces (up to 12 hours) and the inability to combine high translucency with high mechanical properties restricted the use of monolithic zirconia.^[19]but the development of high-speed sintering machines now allows us to mill and sinter monolithic zirconia as a chair-side restoration in just a few minutes.

Translucency is of the most important optical properties needed for dental restorations in the aesthetic area and biaxial flexure strength gives an overall idea about the mechanical properties of the restorations, hence the need to evaluate the effect of the shorter sintering on these properties. The microstructure of the material, which is determined during sintering influence both the translucency and flexural strength of the material. The thickness and grain size affect the material's translucency which tends to decrease as the grain boundaries increase.^[20]Translucency also decrease if some inclusions or vacancies allow for more scattering and refraction and also affect the flexural strength negatively, due to the decreased bonding between grains. Higher flexural strength is achieved with a smaller grain size.^[21, 22]

In the current study, 45 inCoris TZI translucent zirconium oxide discs were prepared and randomly distributed into three groups to be milled with

different speeds. The TP values were then calculated for each specimen as well as the biaxial flexure strength.

According to the results presented in the previous section, the stated null hypothesis was confirmed for the biaxial strength but rejected for the translucency. Accordingly, The ANOVA test showed a significant difference in the translucency of the zirconia after being sintered using different furnaces.

However, there was no significant difference in the biaxial flexure strength. This was in agreement with **Hjerpe et al (2009)**^[23] who reported that variation in sintering time did not influence the mechanical properties of Y-TZP zirconia and **Li et al (2018)**^[24] who stated that dental zirconia showed similar bending strength, hardness and fracture toughness when sintered for 20 min as with conventional sintering time.

Group (S) showed the highest TP values, followed by the control group (CV), while the (SS) group exhibited the least TP, concluding that the latter is the most opaque of the three groups. This is in agreement with **Jansen et al (2019)**^[19] who stated that the translucency decreased with super-speed sintering cycles.

On the contrary, the results of **Kim et al (2013)**^[1] concluded that translucency was inversely proportional to the sintering time; as shorter sintering cycles resulted in smaller grain size and more light transmission. On the other hand, the here-discussed study used a different type of zirconia, and the translucency was measured by TP value and not light transmittance like in Kim et al. This difference in measurement techniques may be the main reason of the dissimilar results in literature.

The higher translucency from the speed sintering compared to super-speed sintering might suggest that the microstructure of the zirconia is negatively impacted by the super-speed cycle, regarding its optical properties. However, further studies comparing grain size, inclusions, and inter-particle spaces are required to determine the reasons why speed sintering created the best translucency.

Therefore, during this experimental study, it was revealed that the speed sintering is the best option available nowadays, since it provides same day delivery restorations, with equal mechanical properties to those sintered using the conventional technique, without hindering the optical properties. However, super-speed systems still remain the only chair-side solution to zirconia restorations since they are small in size, and require only 10-30 min to finish sintering any crown or FPD restoration, unfortunately this comes with the price of reduced aesthetics.

CONCLUSION

The sintering time and speed did not affect the biaxial flexure strength of Y-TZP. As for the translucency, it was the best after performing the speed cycle, followed by the conventional sintering cycle, and the zirconia was least translucent after being sintered in the super-speed cycle. Accordingly we conclude that this chair-side regimen can only be used for non-aesthetic zone restorations. While aesthetic zirconia restorations must still be sintered at a slower speed.

REFERENCES

- Kim, M.-J., et al., Effects of the sintering conditions of dental zirconia ceramics on the grain size and translucency. *The journal of advanced prosthodontics*, 2013. 5(2): p. 161-166.
- Yenisey, M., D.Ö. Dede, and N. Rona, Effect of surface treatments on the bond strength between resin cement and differently sintered zirconium-oxide ceramics. *Journal of prosthodontic research*, 2016. 60(1): p. 36-46.
- Fischer, J., B. Stawarczyk, and C. Hämmerle, Flexural strength of veneering ceramics for zirconia. *journal of dentistry*, 2008. 36(5): p. 316-321.
- Tsalouchou, E., et al., Fatigue and fracture properties of yttria partially stabilized zirconia crown systems. *dental materials*, 2008. 24(3): p. 308-318.
- Vichi, A., et al., Color related to ceramic and zirconia restorations: a review. *Dental materials*, 2011. 27(1): p. 97-108.
- Rues, S., et al., Effect of firing protocols on cohesive failure of all-ceramic crowns. *Journal of dentistry*, 2010. 38(12): p. 987-994.
- Clark, D.E. and W.H. Sutton, Microwave processing of materials. *Annual Review of Materials Science*, 1996. 26(1): p. 299-331.
- Ebeid, K., et al., Effect of changes in sintering parameters on monolithic translucent zirconia. *Dental materials*, 2014. 30(12): p. e419-e424.
- Volpato, C.Â.M., et al., Application of zirconia in dentistry: biological, mechanical and optical considerations, in *Advances in ceramics-electric and magnetic ceramics, bioceramics, ceramics and environment*. 2011, InTech.
- Kaizer, M.R., et al., Speed sintering translucent zirconia for chairside one-visit dental restorations: optical, mechanical, and wear characteristics. *Ceramics international*, 2017. 43(14): p. 10999-11005.
- Rekow, E.D., et al., Factorial analysis of variables influencing stress in all-ceramic crowns. *Dental Materials*, 2006. 22(2): p. 125-132.
- Ersoy, N.M., et al., The effects of sintering temperature and duration on the flexural strength and grain size of zirconia. *Acta biomaterialia odontologica Scandinavica*, 2015. 1(2-4): p. 43-50.
- Johnston, W.M., T. Ma, and B.H. Kienle, Translucency parameter of colorants for maxillofacial prostheses. *International Journal of Prosthodontics*, 1995. 8(1).
- Lim, H.-N., B. Yu, and Y.-K. Lee, Spectroradiometric and spectrophotometric translucency of ceramic materials. *The Journal of prosthetic dentistry*, 2010. 104(4): p. 239-246.
- Chen, Y.-M., et al., Translucency and biaxial flexural strength of four ceramic core materials. *Dental Materials*, 2008. 24(11): p. 1506-1511.
- Schatz, C., et al., Influence of specimen preparation and test methods on the flexural strength results of monolithic zirconia materials. *Materials*, 2016. 9(3): p. 180.
- Pekkan, K., Effect of sintering regimes and thickness on optical properties of zirconia ceramics for dental applications. *International Journal of Applied Ceramic Technology*, 2021.
- Pekkan, G., et al., Factors affecting the translucency of monolithic zirconia ceramics: A review from materials science perspective. *Dental materials journal*, 2020. 39(1): p. 1-8.
- Jansen, J.U., et al., Impact of high-speed sintering on translucency, phase content, grain sizes, and flexural strength of 3Y-TZP and 4Y-TZP zirconia materials. *The Journal of prosthetic dentistry*, 2019. 122(4): p. 396-403.
- Stawarczyk, B., et al., The effect of zirconia sintering temperature on flexural strength, grain size, and contrast ratio. *Clinical oral investigations*, 2013. 17(1): p. 269-274.
- Wang, F., H. Takahashi, and N. Iwasaki, Translucency of dental ceramics with different thicknesses. *The Journal of prosthetic dentistry*, 2013. 110(1): p. 14-20.
- Stawarczyk, B., et al., Three generations of zirconia: From veneered to monolithic. Part I. *Quintessence international*, 2017. 48(5).
- Hjerpe, J., et al., Effect of sintering time on biaxial strength of zirconium dioxide. *dental materials*, 2009. 25(2): p. 166-171.
- li, I., Y. Qiu, and W. Si, Super-Speed Sintered Dental Zirconia for Chair-Side One-Visit Application. *Austin J Nutri Food Sci*, 2018. 6(2): p. 1105.