Original Article

The Effect of Sintering Program on the Compressive Strength of Zirconia Copings

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ABSTRACT

Statement of the Problem: Considering the limitations of conventional sintering of zirconium oxide (ZrO_2) copings, shortening the sintering time can be proposed as an alternative method for making the copings.

Purpose: This study aimed to assess the effects of sintering time on compressive strength of Yttria Tetragonal Zirconia Polycrystal (Y-TZP) copings.

Materials and Method: Thirty copings of pre-sintered 3Y-TZP blanks were milled and sintered in a special furnace for three different durations (n=10 per group). The sintering time was 1 h 15 min for IPS e.max ZirCAD, 4 h 20 min for Speed ZrO_2, and 7 h 20 min for the conventional ZrO_2 group. The specimens were cemented on the brass dies by using conventional glass ionomer cement. The copings were vertically loaded until fracture by using a universal testing machine. The data were analyzed through one-way analysis of variance (ANOVA) and post hoc test to compare the mean differences of compressive strength yielded in three study groups (α=0.05).

Results: The mean $\pm$ SD of compressive strength value was (3617 $\pm$ 543.54) N for IPS-e.maxZirCAD group, (2663 $\pm$ 508.11) N for Speed ZrO_2 group, and (1662$\pm$ 466.71 N) for conventional ZrO_2 group. There were statistically significant differences among compressive strength values of the tested groups (p< 0.05). The highest compressive strength values were obtained from the IPS e.max ZirCAD group.

Conclusion: Within the limitations of this *in vitro* study, it can be concluded that compressive strength of the zirconia copings is affected by the sintering time. High compressive strength of zirconia copings can be obtained by shortening the sintering time.

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Introduction

Zirconia is being increasingly used in dentistry due to the development in evolution of dental advanced ceramics. Its practical properties as well as high productivity in computer-aided design and computer aided manufacturing (CAD-CAM) systems have promoted it to the category of top quality materials. [1]

Zirconia is a polycrystalline available in three allotropic forms. The monoclinic (M) phase remains stable up to 1170° C. At higher temperatures, it turns to tetragonal (T) phase, which is stable up to 2370°C. The cubic phase is formed in 2680°C, which equals the melting point of zirconia. [2] The tetragonal phase of partially stabilized zirconia (PZS) is metastable at room temperature due to the presence of stabilizers such as Yttrium oxide (Y_2O_3) (2-3 mol%). [3] Thereby, the change of phase from T to M occurs under external forces. Under pressure, this change of phase seals the

cracks and consequently inhibits their development. [4] This feature, called toughening transformation, improves the physical and mechanical properties of zirconia. [5]

The CAD-CAM technologies enable the milling of zirconia into reconstructions with complicated geometries. [1, 6] Currently, soft and hard milling processes are known for zirconia. [6-7] In the former, which is done on green and pre- sintered blanks, the zirconia frameworks are sintered after milling. In that condition, the zirconia surface is free of monoclinic phase unless the restoration is sandblasted. On the other hand, the hard-milled zirconia is done on fully sintered frameworks. Therefore, the milling process is lengthy and difficult, yielding restoration with a considerable monoclinic phase on the surface. [7]

The zirconia-based ceramics are favorably employed in dentistry because of their unique mechanical properties that cannot be found in any other ceramic system. [8] Being the most commonly used type of zirconia ceramic material, Y-TZP has some exclusive properties such as high fracture strength, high thermal resistance, low thermal conductivity, and chemical stability. [9] These properties make Y-TZP a quite suitable material for posterior prostheses. The fracture strength is affected by various factors such as size and distribution of the crystalline phase, crown dimensions, geometry, as well as other factors related to some properties of the supporting structure such as the modulus of elasticity. [10-12]

The relationship between the microstructure and mechanical properties of Y-TZP has been discussed in many studies. They all found that the transformation toughening in these ceramics depended on the grain size. [13-15] In the zirconia sintering process, alteration in sintering time and temperature can influence the grain size. [16] The higher the temperature and time is, the larger the grains size would be. [17] The larger tetragonal grains are prone to undergo stress-induced transformation to a balanced structure, resulting in an increased toughness. The maximum toughness is quite close to the critical grain size (1μm). Larger grain size than this critical size leads to spontaneous T-M transformation, which consequently decrease the material stability. [18] Accordingly, lower temperature and time reduce the grain size down to 0.2μm, at which T-M transform does

not occur, and consequently the toughness would be in the lowest level. [19] Contrary to the toughness, the strength is at the highest level in small grains since the fracture size increases in relation to the grain size. [20]

Gupta *et al.* [21] reported that fine grain ZrO_2 (generally<0.5μm) with small concentrations of stabilizing Y_2O_3 contained up to 98% of the post-sintering metastable T phase. High strength coincided with high tetragonal phase content and low strength coincided with high monoclinic phase content.

Ruiz and Ready [16] reported that the grain size increased with increasing the sintering temperature, which in turn, led to increased fracture toughness owing to larger transformation zones. However, they observed no significant difference in biaxial flexural strength of zirconia ceramics with various grain sizes. In contrast, however, Casellas *et al.* [22] found that decreasing the grain sizes lightly increased the flexural strength.

Evidence is limited about the efficacy of short time sintering on the compressive strength of zirconia copings; thus, the objective of the present investigation was to describe the relationship between the sintering time and the compressive strength of zirconia copings. The null hypothesis was that there would be no difference between the compressive strength of 3Y-TZP copings sintered in conventional and short time condition.

Materials and Method

In this *in vitro* study, 30 CNC machined brass dies with 7 mm height, 6° occlusal convergence, and a 90-degree shoulder of 1mm wide finish line were designed and prepared in a lathe (CNC 350; ArixCo, Tainan Hesin, Taiwan). An anti-rotational design was included in the axial surface to ensure repeatable seating of the copings on the dies. The dies were covered with scan spray and scanned by using a 3D laser scanner (3Shape D810; 3Shape, Copenhagen K, Denmark). The scanned data were converted into CAD data (3Shape CAD Design Software; 3Shape, Copenhagen K, Denmark). The size of all machined products was designed to be 20% larger than the dies with due consideration of the sintering shrinkage. No cement space was included for the margin; 30μm was used for the axial and occlusal surface of the abutment. Thickness of the coping was 0.5mm. The design data were converted into processing data and sent to the processing machine (in Lab MCXL; Sirona).

The designed copings were milled from three types of pre-sintered zirconia blanks (Ivoclar Vivadent, Germany) in different sintering programs as displayed in Table 1.

The copings were inspected for any imperfection and rejected in case of any defect. Then, they were cemented conventionally with glass ionomer cement (GIC; Fuji I, GC, Japan) on the brass dies, which were already cleaned with steam and alcohol. All copings were filled with the luting cement and, then, loaded with a vertical force of 10 N for 10 minutes in a cementation device. The copings were clamped in the holder of a universal testing machine (Zwick Z2.5; Zwick, Ulm, Germany). They were vertically loaded on the occlusal surface at a crosshead speed of 0.5mm/m. The minimum force leading to fracture was recorded for each sample. The universal testing machine was controlled via a computer software system, which completed the stress-strain diagram.

Table 1: Classification of the copings to the groups according to sintering time

Group	N	Sintering temperature	Sintering time
Conventional ZrO2	10	1530	7 h 20 min
Speed ZrO2	10	1530	4 h 20 min
IPS-emaxZirCAD	10	1530	75 min

The statistical analyses were performed by using SPSS software, version 14(SPSS Inc.). The mean values and standard deviations (SD) were calculated for each group. One-way ANOVA and post hoc tests were used to compare the results between the groups. The significance level was set at 0.05.

Results

Table 2 represents the mean values and SD of the force leading to fracture in each group. The mean ± SD fracture load was 1662±466.71 in the conventional ZrO2 group, 2663±508.11 in the Speed ZrO2 group, and 3617±543.54 in IPS e.max ZirCAD group.

Table 2: The mean values and standard deviation of fracture load in Newton

Group	Mean	SD	MIN	MAX	*p* Value
IPS emax Zir CAD	3617	543.54	3060	5020	
Speed ZrO2	2663	508.11	2000	3520	<0.05
ConventionalZrO2	1662	466.71	1100	2650	

The results of post hoc test showed that compressive strength was significantly different among the test groups (df=2, F=17.488, *p*= 0.000)

The highest mean± SD of fracture load was observed in IPS e.max Zir CAD (3617± 543.54) which was sintered with in the shortest sintering time (75min). The results of ANOVA and post hoc indicated that the compressive strength was affected by the sintering time.

Discussion

This study aimed to evaluate the relationship between sintering time and compressive strength in 3Y-TZP copings. The findings rejected the null hypothesis through proving that the sintering time significantly affected the in vitro compressive strength of cemented zirconia copings ($p< 0.05$).

The mean compressive strength in the present study was found 3617, 2663, and 1662 N for groups 1 to 3, respectively. It is difficult to compare failure loads found in the literature to those found in this study, due to different experimental variables. A study reported a failure load of 381 N for zirconia coping of 0.6 mm thickness on non-carious incisor. [23] In another study, the failure load was 1670 N in zirconia coping with 0.4mm thickness on resin die. [24] Other studies surveyed the failure load in complete crowns and reported the fracture load values to range between 980 and 1400 N. [25-26] In the present study, the compressive strength was assessed on brass dies instead of natural teeth to control the accuracy and certainty of reposition and similarity of parameters such as margin and convergence coping. Thus, the copings geometries were identical, although their elastic modulus and fracture strength were not similar to natural teeth. The results of some previous studies showed that increasing the elastic modulus of supporting structure led to an increase in the failure resistance of the crown. [27-30] It can be concluded that the higher range of failure load in this study was due to the high elastic modulus of the supporting structure, which may lead to an overestimation of the clinical values.

Zirconia crowns can bond with conventional protocol or resin-bonded cements. [31] Studies have reported higher fracture strength in ceramic restorations cemented with adhesive cement compared with retentive cements. [32-33] The study of Rekow *et al.* [34] and

Zesewitz *et al.* [35] have shown that there was not a substantial difference in fracture load between zirconia crowns that were cemented with resin-bonded cements and metal dies that were cemented with glass ionomer cement. They concluded that the strength of zirconia might overcome against the influence of cement properties and thickness. In the current study, the glass ionomer cement was used with minor self-adhesive properties. In the present study, we surveyed the zirconia copings without veneering materials, because the recent studies reported that the presence and thickness of veneering porcelain affected the compressive load to failure. [25-26, 36] Moreover, the copings were fabricated with flat occlusal morphology because the natural occlusal anatomy was likely to affect the outcome due to the lateral component of the loading force. [37]

The relationship between the grain size and the mechanical properties in 3Y-TZP was previously studied. It was found that the grain size determined the transformation toughening effect, toughness, and strength. [38] Sintering condition directly influenced the grain size of zirconia. The strength of porous ceramics decreases significantly with the increase of porosity. The compressive strength is influenced by the porosity and pore size. [39] Kim *et al.* [40] evaluated the effects of sintering time on the grain size of zirconia. They reported that the density of all samples ranged from 6.06 to 6.07 g/cm. They noted no statistically significant difference among the samples sintered for different time spans. However, Tekeli and Erdogan [41] noticed that the sintering time influenced the density and mechanical properties. They reported that high sintering temperature and extended sintering time increased the grain size and consequently, the number of pore; and thereupon yielded a material with reduced mechanical properties. In contrast, Hjerppe *et al.* [42] showed that short sintering time for zirconia decreased the grain size. Yet, the results were not statistically significant and had no effect on the mechanical properties. Cottom *et al.* [19] reported that shorter sintering time yielded smaller grain size.

In the current study, different sintering time was applied to pre-sintered zirconia specimens in order to obtain dense sintered copings with different grain size, based on our finding the shorter sintering time resulted higher compressive strength. However, the strength of ceramic material depends more upon the experiment condition rather than the materials properties. In contrast, the fracture toughness is a more inherent property of ceramics, which is not affected by surface flaws and the initial crack size. [43] Based on Hall-Petch [44] regime, the fracture in large grain sizes is controlled by the intrinsic defects; where as in small grain sizes, failure is governed by extrinsic processing defects such as preparation cracks and pores. In this case, the grain size is much smaller than the defects. Thus, the fracture strength depends on the grain size less than it does on the geometry of the fracture origin. Hoffman *et al.* [45-46] demonstrated that the critical stress for microcrack formation decreased with increasing the grain size. They also explained that the failure in grains smaller than a certain critical size occurred due to the stress concentrations around the pore before the formation of the microcracks. The criterion for fracture changes from limited crack extension to limited crack initiation. This variation increases the fracture strength in smaller grains. According to the result of the present study, shortening the sintering time could led to smaller grain size in the IPS e.max ZirCAD group, which in turn increased the compressive strength load value.

This study has some limitations; short time sintering may also influence other properties of the zirconia ceramics but this study was limited to only one feature. Static *in vitro* tests were used in current study; however, dynamic fatigue tests are more representative of clinical masticatory forces and further *in vitro* and *in vivo* tests are required.

Conclusion

Within the limitations of this study, it can be concluded that the sintering protocol affects the compressive strength of zirconia copings, the shorter the sintering time, the higher the compressive strength.

Conflict of Interest

The authors declare that they have no conflict of interest.

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