

Effects of sintering temperature and particle size on the translucency of zirconium dioxide dental ceramic

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Abstract The aim of this study was to evaluate the effects of sintering temperature and particle size on the translucency of yttrium stabilized tetragonal zirconia polycrystals (Y-TZP) dental ceramic. Eighty disc-shaped and cylindrical specimens were fabricated from zirconia powers of particle size 40 and 90 nm. These specimens were sintered densely at the final sintering temperature 1350, 1400, 1450 and 1500°C, respectively. The visible light transmittance, sintered density and microstructure of the sintered block were examined. The results showed that the sintered densities and transmittances increased with the temperature from 1,350 to 1,500°C. Y-TZP could gain nearly full density and about 17–18% transmittance at the final sintering temperature of 1,450–1,500°C. The 40-nm powders had higher sintered density and transmittance than the 90-nm. The translucency of Y-TZP dental ceramic could be improved by controlling the final sintering temperature and primary particle size.

1 Introduction

With the widespread use of ceramic restorations, all-ceramic materials have been advocated as the better choice for their excellent biocompatibility and superior optical properties [1, 2]. The optical property of core materials plays an

important role in matching the ceramic restorations to the natural appearance of teeth. Kelly et al. [3] indicated that core translucency was one of the primary factors in achieving esthetics and affected the shade of the artificial restorations greatly.

Among a variety of core materials partially stabilized zirconia has been proved to possess better mechanical properties due to its transformation toughening [4–7]. It has been regarded as “Ceramic Steel” [8] and been clinically used to fabricate esthetic single crowns, three-unit bridge or even multi-unit bridge in the posterior tooth segment [9]. However, zirconia-containing core materials have poor translucency and are difficult to satisfy esthetic requirement. Heffernan et al. [10] and Chen et al. [11] reported that In-Ceram Zirconia containing 33% partially stabilized tetragonal zirconia polycrystals had a higher contrast ratio compared to other all-ceramic materials. Moreover, because the contrast ratio is closed to that of the metal-ceramic restoration, both materials have been found to be completely opaque. Therefore, it is essential to enhance its translucency for the esthetic requirement.

Translucency is defined as the relative amount of light transmission or diffuse reflectance from a substrate surface through a turbid medium [12]. The translucency of dental ceramic has a close relationship with its chemical composition and microstructure [13]. The chemical nature, the amount of crystals, the size of particles, the pores and the sintered density determine the amount of light that is absorbed, reflected, and transmitted. All of the above influence the optical property of core ceramics [10]. Further, the main factor is multiple scattering of light in the ceramic grains. Multiple crystalline contents used to strengthen the ceramic reduce the translucency because of the different refractive indices and the inhomogeneity of crystals. Since Y-TZP is polycrystals and has a different

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refractive index to the matrix, most of the light passing through it is intensively scattered and diffusely reflected, leading to opaque appearance. So the translucency of zirconia core ceramic is commonly lower than that of spinell, alumina, and feldspathic porcelains. In view of the esthetic drawbacks of zirconia, researchers have worked on optimizing production conditions to improve the optical property [14].

Recently some studies [15, 16] have suggested that microcrystals and full densification can simultaneously enhance mechanical properties and light transmittances. In this study, partially nanostructured powders (ZrO_2 -3 mol.% Y_2O_3) were densely sintered with microcrystals to achieve the high translucency. These zirconia blocks for CAD/CAM (Computer-aided design/Computer aided manufacture) are made from nanopowders and can meet the demands of esthetics and strength when used for dental restorations [14]. The mechanical property of zirconia core has been studied in many aspects, but few studies have focused on its translucency.

In the fabrication procedures many factors influence the translucency of yttrium partially stabilized zirconia, such as primary particle size, heating rate, sintering temperature and atmosphere of sintering. Among these factors, the final sintering temperature has a major impact on the densification processes and microstructure of Y-TZP nanopowders. The relationship between the different sintering temperature and the transmittance has not been reported before. So this study evaluated the effects of the sintering temperature and the particle size on the light transmittance to find out the method of improving the translucency of Y-TZP dental ceramic.

2 Materials and methods

2.1 Specimen preparation

Two kinds of nano-sized zirconia powders partially stabilized with 3 mol.% yttria were used in this study. One is TZ-3YB-E of reported particle size around 40 nm and the other is TZ-3YSB-E of around 90 nm (Tosoh Corporation, Tokyo, Japan). Forty disc-shaped specimens, 15 mm in

diameter and 0.50 ± 0.01 mm in thickness, were fabricated from both powders. Approximately 0.48 g powders were used in preparation of a 0.5-mm-thick disc. The powders were processed in a custom-made, cylindrical metal mold with a 15 mm inner diameter, subsequently pressed by the powder machine (Tianjin KeQi High and New Technology Co, Tianjin, China) under 20 MPa for 30 s, and finally were compacted under 200 MPa for 1 min by cold isostatic pressing machine (Chuan-xi Machine Co, Ltd, Sichuan, China). Forty cylindrical specimens of 5.70 g powders were made to measure the sintered density according to the above procedure.

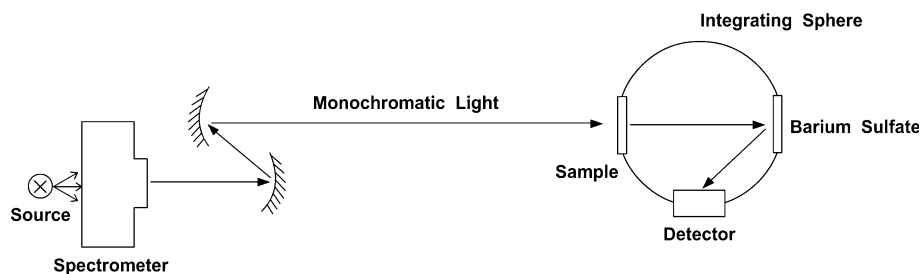
These specimens were sintered in a high-temperature furnace (Nabertherm Industrial Furnaces Co. Ltd., Germany) and were divided into four groups based on different sintering temperatures 1350, 1400, 1450 and 1500°C respectively. Each group included five discs and cylindrical specimens. Specimens were embedded with alumina refractory beads in the alumina crucible and sintered in accordance with following protocol: heated from room temperature to final temperature at a rate of 200°C/h, held for 2 h and naturally cooled to room temperature again. Between 200 and 800°C, the oxygen was supplied to assure complete combustion of organic matter in the powders.

The disc-shaped specimens were finished flat on a metal-bonded diamond disk to guarantee the uniform thickness of 0.50 mm and were polished on both sides by diamond polishing paste of a sequential grit size 25, 10, 5 and 1 μ m respectively to achieve the smooth surface. The thicknesses of these specimens were measured with a digimatic caliper (Mitutoyo Corp, Kawasaki, Japan) after ultrasonically cleaned in distilled water for 15 min. Measurements were repeated three times and averaged. Only those specimens with the thickness of 0.50 ± 0.01 mm were included in this study.

2.2 Visible light transmittance measurement

The transmittance of specimens was measured by spectrophotometric arrangements (Fig. 1). The spectrophotometer consisted of two instruments: a spectrometer with a double-prism monochromator (Bentham Instruments Ltd., England) to produce the light of any selected wavelength, and a

Fig. 1 Schematic illustration of apparatus for measuring spectral transmission



photometer with a silicon photodetector (EG&G Co, USA) to measure the intensity of light. The instruments were arranged so that specimens could be placed between the spectrometer beam and the photometer. And the specimen was placed at the entrance port of the integrating sphere in order that the total amount of light transmitted and scattered through it could be measured. Then the photometer delivered a voltage signal to a galvanometer and the number was transmitted to a computer for further manipulation.

The instrument was preheated for at least 30 min and was calibrated prior to use. The reflection spot was adjusted to assure the incidence of light vertical to the specimen surface and to align the light source and specimen. The specimens were measured with 0° illumination and diffuse viewing geometry. The light from tungsten halogen lamp was guided through a monochromator, which picked the light of one particular wavelength out of the continuous visible light spectrum before it passed through the specimen. Finally the intensity of remaining light was gathered by the integrating sphere and measured by the photometer. The transmittance (*T*) for this wavelength was approximated as the following formula: $T = (I/I_0) \times 100\%$, where I_0 was the intensity of the incident beam, and *I* the intensity passing through the specimen. Based on the formula, the spectral integral transmittance which demonstrated the ratio of the visible light flow could be obtained.

The size of the measuring port was a diameter of 4 mm and the working wavelength ranged from 400 to 780 nm of the visible light spectrum, allowing for measuring every 10 nm. The center of every specimen was measured three times. All measurements were performed by the same operator in a dark room, with the working temperature of 18–25°C.

2.3 Sintered density measurement

The sintered density of the cylindrical specimens under the different sintering temperature was determined by Archimedes' method. The sintered density was calculated according to the following equation:

$$\rho = \frac{W_D \times \rho_W}{W_S - W_{SS}}$$

W_D is the dry weight of the sintered specimen, W_{SS} the saturated suspended weight of the specimen in the water at room temperature, W_S the saturated weight of the specimen after removing water from the surface, ρ_W the density of water at 20°C (0.9982 g/cm³ used in this study).

One specimen was measured three times and the sintered density value was determined by the average of the five pieces. The relative density was calculated by the following equation:

$$D = \rho/\rho_0 \times 100\%$$

ρ_0 is the theoretical density of yttrium stabilized tetragonal zirconia polycrystals (6.10 g/cm³ [17]).

2.4 Scanning electron microscopy

Specimens were fractured for examination by field emission scanning electron microscopy (INSPECT F, FEI Co., Eindhoven, The Netherlands). Before examination the fracture surfaces were etched in a strong acid solution for 2 min and then were ultrasonically cleaned in distilled water for 15 min. Pores, grains' shapes and distribution of the fractured surfaces coated with gold were observed.

2.5 Statistical analysis

Two-way analysis of variance (ANOVA) followed by Tukey's multiple-comparison test ($\alpha = 0.05$) was used to analyze the effects of sintering temperature and particle size on the transmittance of yttrium partially stabilized zirconia by SPSS11.0 (SPSS Inc., Chicago, USA) statistic software.

3 Results

The sintered densities of zirconia specimens after sintered under the different final temperature were showed in Table 1. The sintered densities ranged from 6.01 to 6.06 g/cm³ for the 40-nm powders and from 5.72 to 6.05 g/cm³ for the 90-nm. As could be seen from the statistical result, both the sintering temperature and particle size had a significant interactive effect on the sintered densities ($P < 0.001$). Multiple-comparison between groups showed significant differences in the temperature and particle size, too. The sintered densities increased with the temperature

Table 1 Descriptive statistics of sintered densities and spectral integral transmittances of specimens prepared from zirconia powders of particle size 40 and 90 nm at four different sintering temperatures

Particle size	Sintering temperature (°C)	Sintered density (g/cm ³)	Relative density (%)	Spectral integral transmittance (%)
40	1,350	6.0141 ± 0.0062	98.59	15.51 ± 0.58
	1,400	6.0626 ± 0.0071	99.39	17.50 ± 0.47
	1,450	6.0627 ± 0.0074	99.39	17.83 ± 0.16
	1,500	6.0481 ± 0.0045	99.15	18.01 ± 0.07
90	1,350	5.7173 ± 0.0305	93.73	1.76 ± 0.06
	1,400	5.9105 ± 0.0607	96.89	8.82 ± 0.58
	1,450	6.0170 ± 0.0111	98.64	16.98 ± 0.37
	1,500	6.0514 ± 0.0100	99.20	17.58 ± 0.10

from 1,350 to 1,500°C except for the 40-nm powders at 1,500°C (Fig. 2). The densities of the 40-nm powders at 1,400–1,500°C and the 90-nm at 1,500°C were over 99% of the theoretical values. At the same temperature, the sintered densities of the 40-nm powders were higher than that of the 90-nm.

The transmittances of different groups were also showed in Table 1. The transmittances of zirconia specimens ranged from 15.51 to 18.01% for the 40-nm powders and from 1.76 to 17.58% for the 90-nm. Two-way ANOVA revealed that the sintering temperature and particle size had a significant interactive effect on the transmittances ($P < 0.001$). Tukey's post hoc test also indicated that transmittances increased with the sintering temperature and at the same temperature the transmittance of the 40-nm powders was higher (Fig. 3). As the temperature was higher than 1,400°C the zirconia of 40-nm particles gained

the highest transmittance, while the critical temperature for the 90-nm was 1,450°C.

Scanning electron micrographs of zirconia ceramic at the four different temperatures demonstrated the grain microstructure (Figs. 4, 5). The very fine faceted grains were showed. Zirconia specimens from the 90-nm powders sintered at 1,350°C had greater porosity and the pores of irregular shapes were often located at the interboundary regions. With the increasing temperature the zirconia crystal structure became more compact, while porosity, defects and flaws decreased. The grain size ranged from 200 to 400 nm at 1,400–1,450°C. However, at 1,500°C the grains grew unevenly and varied between 400 and 600 nm. It was also found that distinct grain boundary was formed. The grain sizes of the 40-nm powders were significantly smaller than that of the 90-nm.

4 Discussion

Transmittance, a physical parameter representing the ability of light to pass through certain medium [18], is better than other parameters of displaying the ceramic translucency. Thickness is the primary factor relating to the light transmittance [10, 19], as can be verified by the Law of Lambert: $T = e^{-2x}$. It identifies that the transmittance (T) decreases exponentially with the increasing of thickness (x). Moreover, manufacturers and clinical researchers recommend the thickness of zirconia core should be 0.30–0.50 mm to compromise the strength and optical properties [9]. Therefore the thickness of all zirconia specimens in this study was controlled to ensure the uniform dimension. The standard deviation of the transmittance among the specimens of each group was minimal, which allowed for a small sample size ($n = 5$). The results of this study supported the hypothesis that the sintering temperature and particle size could influence the transmittance of Y-TZP.

The sintering temperature determines the properties of ceramics by effect on the microstructure and the crystalline phases [20, 21]. Sintering can eliminate the interparticle pores in a granular material by atomic diffusion driven by capillary forces [16]. As the temperature rises, the particles are sintered together and pores on grain boundaries are reduced by solid-state diffusion. So the sintered density increases. Meanwhile the tendency of transmittance has been mainly consistent with the relative density. It suggests that pores can be the main factor which affects the translucency greatly. Significant difference of the refractive index between the particle and matrix results in obviously scattering [22]. Because the relative refractive index of zirconia (approximately 2.20) [23] and that of pores (1.00) is the highest among all-ceramic core materials, pores

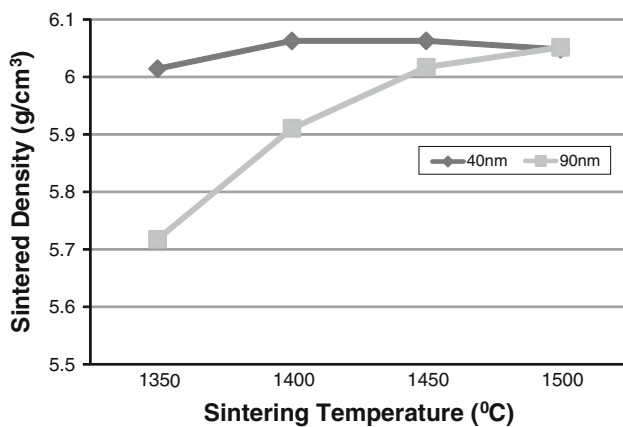


Fig. 2 Sintered densities of specimens prepared from zirconia powders of particle size 40 and 90 nm at four different sintering temperatures

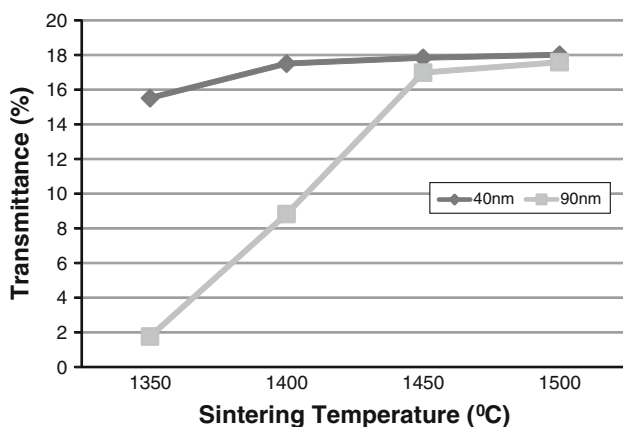


Fig. 3 Transmittances of specimens prepared from zirconia powders of particle size 40 and 90 nm at four different sintering temperatures

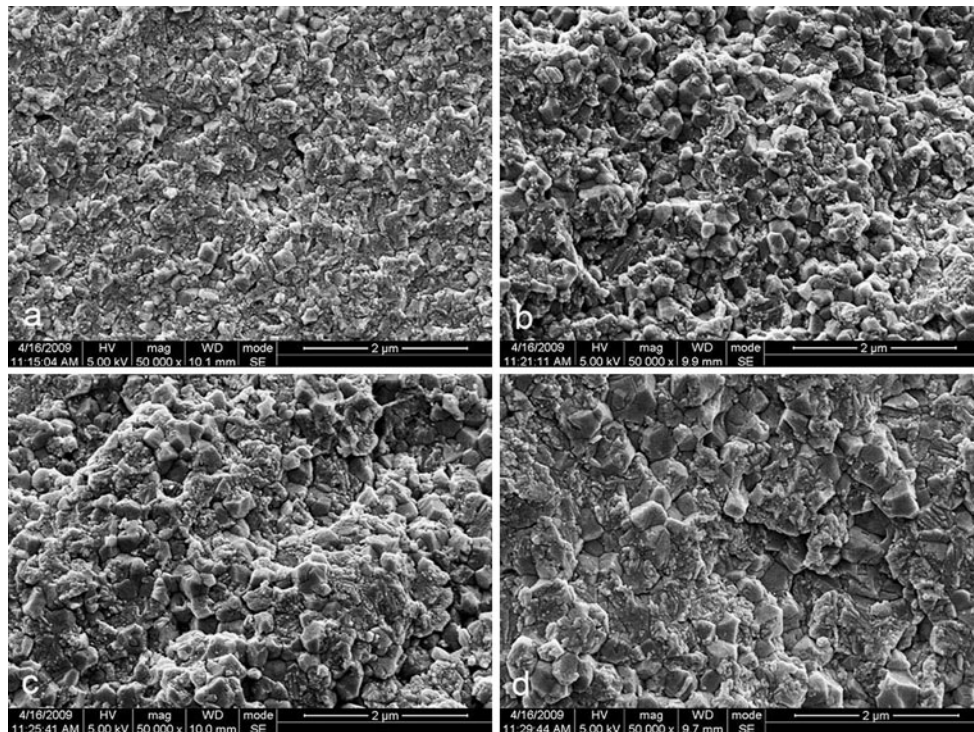


Fig. 4 Micrographs of zirconia specimens from the 40-nm powders sintered at different temperatures. **a** 1,350; **b** 1,400; **c** 1,450; **d** 1,500°C, respectively

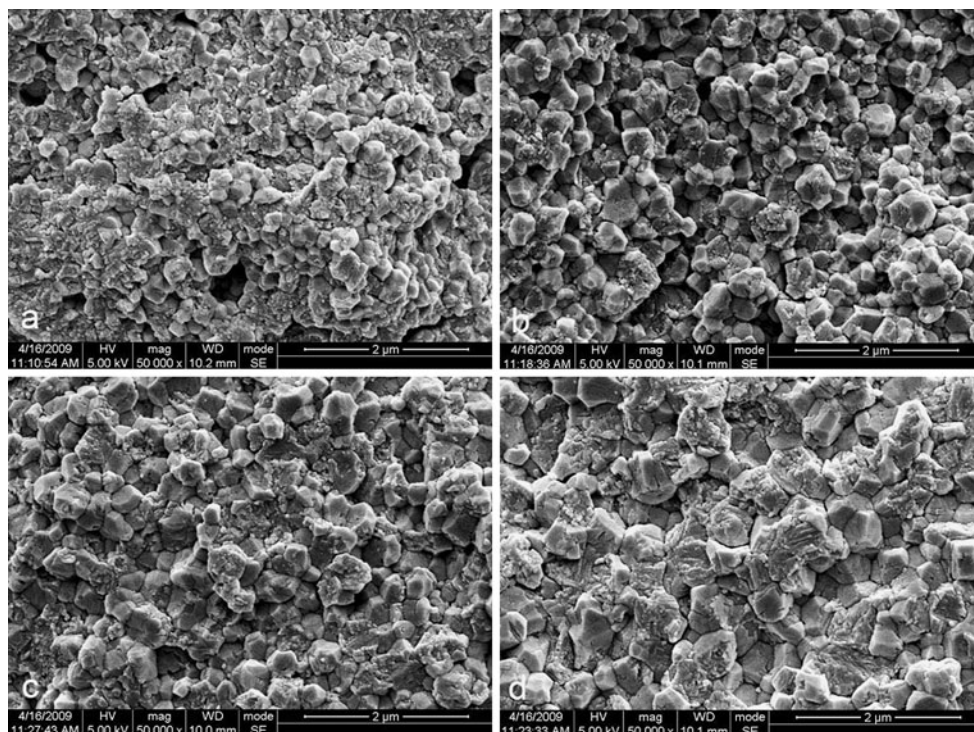


Fig. 5 Micrographs of zirconia specimens from the 90-nm powders sintered at different temperatures. **a** 1,350; **b** 1,400; **c** 1,450; **d** 1,500°C, respectively

become the greatest scattering center in zirconia ceramic. So gaining full density can diminish the porosity and improve the translucency greatly. That was verified by the result of this study. The density of zirconia specimen was over 98.5% of theoretical value at 1,450–1,500°C. Since the high density was achieved, the transmittance has risen to over 17%. The results have suggested that the final sintering temperature for the densification of nanozirconia is 1,450–1,500°C. Many studies [24] also pointed out that pores were highly efficient light scatters and thus very low porosity was required for ceramic to be transparent. It might be an effective way to reduce either the residual porosity or the size of pores by increasing the temperature to an appropriate value.

In this study the transmittance was very low at 1,350°C, especially for the 90 nm, but has risen greatly with only a 100°C temperature increase. As it demonstrated, the relative density increased from 93.73 to 98.64% for the 90 nm at 1,350–1,450°C. The SEM images also identified the fracture surface had obvious pores at 1,350°C but had compact structure at 1,450°C. Even a minor amount of residual porosity could significantly prevent translucency in oxide ceramics [25, 26]. That could explain that the transmittance increased significantly while the relative sintered density increased a little. And it has suggested that the full densification occur mainly at 1,350–1,450°C. In the sintering processes, the final-stage is always accompanied by the rapid reduction of porosity. So the final temperature is essential to achieve the nearly full density and high transmittance.

The micrographs revealed that the temperature as high as 1,450–1,500°C could eliminate the porous area caused by incomplete firing, while overfiring might yield rapid grain growth [22]. It could explain that the relative density of the 40-nm specimen has decreased at 1,500°C. At the temperature higher than 1,450°C, the zirconia could reach the nearly theoretical density and reduce the volume fraction of porosity to a minimum. Besides, the higher transmittance is due to the homogenous structure of tetragonal zirconia microcrystal which could reduce the scattering light at the interface between adjacent crystals. This microstructure has a close relationship with the appropriate temperature and enough time of phase transformation. The monoclinic zirconia transforms into tetragonal phase when the temperature is up to 1,170°C [6]. In the process of increasing temperature, the slow rate and enough holding time at the high-temperature section can be benefit to completely transform and prevent the excessive growth of grains. The addition of stabilizing oxides such as 3 mol.% Y_2O_3 allows the retention of the tetragonal phase at the room temperature. Thus this transformation microstructure determines not only the optical characteristics but also the mechanical properties.

Currently, the final sintering temperature of zirconia ceramics available for dental application varies between 1,350 and 1,550°C depending on the manufacturers [20]. Representative all-ceramic systems of 3Y-TZP include InCeram[®] zirconia (VITA Zahnfabrik), Cercon[®] (Dentsply International), Lava[™] (3 M[™] ESPE[™]), Procera[®] zirconia (Nobel Biocare[™]) and Zirkon[®] (DCS Dental AG). Al-khunaizi R [27] offered a ranking of different core materials according to the contrast ratio (from most translucent to most opaque): InCeram Spinell > Lava, YZ-55 > Cercon, InCeram Alumina > Incoris ZI > InCeram Zirconia. Lava and Cercon with Y-TZP as the chief constitute, had the lowest contrast ratios as 0.69 and 0.77 respectively, which represented the relatively high translucency in core materials. The result of this study has also supported that Y-TZP could gain the high transmittance.

The crystal size of primary particles and the degree of agglomeration have a great effect on the packing achievable in green body, grain size and the final sintered density [28, 29]. The 3Y-TZP powders used in this study consisted of spray-dried agglomerates of nanocrystals. It is well-known that the high surface area of nanoparticles supplies the substantial sintering driving force by which the activation energy is reduced and sintering temperature can be decently lowered [30]. The 40-nm primary particles with the higher surface area, higher sintering activity and short diffusion distances could be easily sintered to the theoretical density and achieve higher transmittance at the relatively lower temperature. So the 40-nm powders had the higher density and transmittance than that of the 90-nm powders at the same sintering temperature. Besides, the 40-nm powders could form the smaller crystals. The volume fraction and mean size of the crystals could influence the transmittance. Grains similar in size to the visible light wavelength (380–780 nm) produce the greatest scattering [14]. Previous results have shown that polycrystalline alumina with smaller crystal sizes are more translucent than those with larger crystal sizes [25]. It was possible that the smaller crystal sizes in this study contributed to the high degree of translucency. Anusavice et al. [31] have also noted that the colorants of ceramic could not affect the crystallization process to control the mean size of grains for the translucency.

Nowadays many studies [15, 32] have introduced translucency to zirconia by consolidating nanopowders to full density with nanocrystals through the industrial sintering technique such as hot-isostatic pressing (HIP), microwave and millimeter wave sintering, spark plasma sintering (SPS). Those zirconia products have been widely used in the industrial field. Meanwhile the fabrication of zirconia dental ceramic could learn from those methods. In addition, the mechanical and physical properties of zirconia core are required to be consistent with translucency for

clinical application. Further study is needed to confirm the relationship among mechanical properties, the sintering temperature and the particle size to improve the sintering procedure for Y-TZP dental ceramic under the pressureless condition.

5 Conclusions

Within the limitations of this study, the sintering temperature and particle size significantly affected the density and transmittance of Y-TZP ($P < 0.001$). Y-TZP could gain nearly full density and 17–18% transmittance at the final sintering temperature of 1,450–1,500°C. Y-TZP of the smaller nanoparticles had higher density and transmittance than that of the larger particles.

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