Strength of 3Y-TZP and feldspathic porcelain subjected to different cooling methods

Antonio Alves de Almeida-Júnior[1], Diogo Longhini[2], Beatriz Regalado Galvão[3], Claudinei dos Santos[4], Gelson Luis Adabo[2]

[1]Universidade Tiradentes – UNIT, School of Dentistry, Department of Oral Rehabilitation, Aracaju, SE, Brazil
[2]Universidade Estadual Paulista – UNESP, School of Dentistry, Department of Dental Materials and Prosthodontics, Araraquara, SP, Brazil
[3]Universidade Potiguar - UnP, School of Dentistry, Department of Oral Rehabilitation, Natal, RN, Brazil
[4]Universidade do Estado do Rio de Janeiro – UERJ, Resende School of Technology, Department of Engineering Materials, Resende, RJ, Brazil

Abstract

Aim: To investigate the effect of the cooling rate on flexural strength of monolayer and bilayer porcelain/zirconia (Y-TZP) bars. **Methods:** Forty-five specimens were made for each design group: (PM) monolithic specimens of veneer porcelain Vita VM9 (Vita, Germany); (ZM) monolithic specimens of zirconia (ZiHP; ProtMat, Brazil); (PB) bilayer specimens zirconia/porcelain with porcelain on lower surface; and (ZB) bilayer specimens porcelain/zirconia with zirconia on lower surface. Each group was cooled by three different methods after porcelain sintering: slow – specimens were cooled inside the turned-off furnace; normal – specimens were removed from the furnace and cooled in air at room temperature; and fast – specimens were removed from the furnace at 910°C and cooled by compressed air for 10 s. Specimens were polished and flexural strength was measured in water at 37 °C (n=15). Maximum load at fracture was recorded, and equations for simple (monolayer) and composite (bilayer) structures were used to calculate the flexural strength. Results were analyzed using one-way ANOVA (p<0.05) and Tukey test separately for each design. **Results:** The results of one-way ANOVA were statistically significant only for the PB group. The post-hoc Tukey test showed the highest flexural strength for fast cooling and the lowest for slow cooling; the normal cooling was statistically similar to both. **Conclusions:** Cooling methods affected only the flexural strength of bilayer specimens with porcelain on low surface (under tension) when the slow cooling method was used.

Keywords: dental porcelain; zirconium; material resistance.

Introduction

Medical applications of ceramic system based on Yttria-stabilized zirconia (3Y-TZP) have increased due to its biological and mechanical properties and improvement in computer-aided design/computer-aided manufacturing (CAD/CAM) technology. Zirconia was introduced into the dental market as framework for ceramic fixed partial dentures due to its sufficient strength and toughness, and it is believed to resist the masticatory forces in posterior teeth, comparable to several metal-ceramic alloys[1]. However, high crystalline zirconia is an opaque material, and, for esthetical reasons, 3Y-TZP frameworks have to be veneered with specific feldspathic dental porcelains[2].

Nevertheless, the veneer porcelain is the weakest part of this system due to its low content of crystalline phase. Moreover, this system seems to behave

differently from conventional metal-ceramic prostheses. After three years of service time, chipping of zirconia porcelains is described to have a failure rate of 15%, while this rate for metal-ceramic prostheses is less than 0.5% for crowns and 3% for fixed partial denture in five years[3-6].

Among the reasons for failure, residual tension is the most discussed issue in the literature[7-11]. Incompatible thermal expansion coefficient[7], non-uniform porcelain thickness[12], inadequate substructure design[13], low thermal conductivity of zirconia, fast cooling rates[11], and intrinsic strength of these ceramic materials[14-15] may develop tension stress in the system and induce the occurrence of failures in 3Y-TZP/porcelain prostheses[10,15]. Moreover, zirconia and metal frameworks behave differently after porcelain sintering. During the cooling, an excessive compressive stress may be compensated by plastic flow or thermal creep of the metal, while similar compensation is not possible in 3Y-TZP frameworks because of its high rigidity. Therefore, veneering materials for all-ceramic system should have high mechanical strength[16].

Different cooling rates after porcelain firing schedule have been proposed for reducing those residual stresses[10,17-18]. The study of flexural strength of veneering porcelains and zirconia using faster or slower cooling is an important parameter to improve the clinical behavior and performance of veneered zirconia prostheses. The aim of this study was to investigate if cooling methods change the flexural strength on monolayer and bilayer specimens of zirconia and feldspathic porcelain subjected to different cooling methods. The null hypothesis was that the cooling method did not modify the flexural strength of monolayer and bilayer zirconia/porcelain specimens.

Material and methods

A three-point flexural strength test was performed in four different designs (Figure 1) of monolayer or bilayer bar specimens of 3Y-TZP (ZiHP; ProtMat Materiais Avançados, São Paulo, SP, Brazil) and feldspathic porcelain (VITA VM9; VitaZahnfabrik, Bad Säckingen, Germany) subjected to three different cooling methods.

Forty-five specimens of each design were prepared according to the dimensions recommended by ISO 6872:2008 for a three-point flexural strength test (4 ± 0.25 mm in width, 1.2 ± 0.2 mm in thickness and 22 mm in length). The

thickness of bilayer specimens was in a ratio of 1:1. Zirconia specimens were cut approximately 25% larger than the final dimensions with a diamond disk (15LC; Buehler Ltd, Lake Bluff, IL, USA) in a precision saw (ISOMET 2000; Buehler Ltd.) at low speed. After cutting, the specimens were ground using 600-grit silicon carbide (SiC) abrasive papers (Norton Abrasivos, São Paulo, SP, Brazil) on a mechanical polisher (Buehler Metaserv 2000; Buehler UK Ltd., Coventry, England) under running water. The specimens were sintered in a $MoSi_2$ oven (INTI FE 1800; Maitec, São Carlos, SP, Brazil) at 1.530°C for 2 h, heating rate of 8 °C/min, and cooling rate of 5 °C/min, according to the manufacturer's recommendation. The firing schedules for zirconia and porcelain are shown in Table1.

Porcelain specimens were made by mixing VM9 powder and VITA Modeling Liquid (VitaZahnfabrik). Slurry was prepared and condensed into polyether mold (Impregum F; 3M ESPE, Seefeld, Germany) 20% larger than the final dimensions, to compensate for porcelain contraction. After the excess liquid had been soaked up with an absorbent tissue, the specimens were fired in an oven for ceramics (Aluminipress; EDG, São Carlos, SP, Brazil) according to the recommendations of the porcelain manufacturer (Table 1).

Table 1. Firing schedules of the materials

Step	Zirconia	Base Dentine Washbake VM9	Base Dentine VM9
Pre-Drying Time (min)	-	2	6
Pre-Drying Temperature (°C)	-	500	500
Heating Rate (°C/min.)	8	55	55
Firing Temperature (°C)	1530	950	910
Holding Time (min)	120	1	1

For bilayer specimens, the porcelain manufacturer recommends the base dentine washbake firing previous to dentine porcelain. Washbake porcelain was applied on one side of the zirconia bilayer samples and fired according to the manufacturer's instructions (Table 1). After the washbake layer cooling, dentine porcelain was applied as previously described.

After porcelain firing (groups PM, PB, and ZB) or porcelain firing simulation for the ZM group, three cooling methods were performed (n = 15): *slow* – samples were left inside the closed turned-off furnace until it reached the room temperature; *normal* – the elevator of the furnace was down,
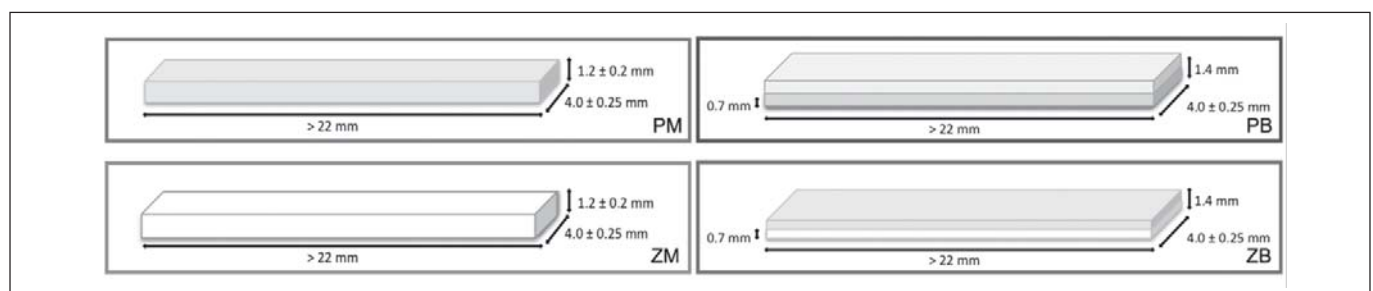

Fig. 1. Design groups: (PM) monolithic specimens of porcelain; (PB) bilayer specimens 3YTZP/VM9 with porcelain in lower surface; (ZM) monolithic specimens of 3Y-TZP; (ZB) bilayer specimens VM9/3Y-TZP with zirconia in lower surface

and when the temperature inside the furnace reached 500°C, the samples were removed and cooled in air at room temperature; *fast* – samples were blasted directly by compressed air immediately after removal from the furnace. When cooling was finished, the porcelain in the specimens were grounded and polished using 120-, 220-, 320-, 400-, 600-, and 1200-grit SiC abrasive papers (Norton Abrasivos Brasil, São Paulo, SP, Brazil) on a mechanical polisher (Buehler Metaserv 2000; Buehler UK Ltd.) under running water. Bilayer specimens were randomly allocated to the design group.

The three-point flexural strength test was performed in a universal testing machine (DL 2000; EMIC, São José dos Pinhais, PR, Brazil) with a 5.0 kN load cell and at crosshead speed of 1.0 mm/min until failure. The specimens were placed in the sample holder, which had a span of 15 mm between the two 0.8 mm radius rounded bearers and loaded by a 1.6 mm radius rounded steel knife edges. Testing was carried out in distilled water at 37 °C with the load applied at the midpoint of the samples. The flexural strength was calculated for the monolayer specimens according to Equation 1.

$$\sigma_f = \frac{3FL}{2wh^2} \qquad \text{Eq 1}$$

Where σ is the maximum center tensile stress (MPa), F is the load at fracture (N), L is the distance of the two supports (mm), w is the width of the specimen (mm), and h is the height of the specimen (mm).

For bilayer specimens, flexural strength was calculated using Equation 2, where σ_f is the maximum center tensile stress (MPa), L is the distance of the two supports (mm), P is the load at fracture (N), E_t is the Young modulus (according to the manufacturer) of material under tensile stress (GPa), t_t is the height of the material under tensile stress (mm), E_c is the Young modulus (according to the manufacturer) of material under compression (GPa), t_c is the height of the material under tensile stress (mm), and w is the width of the specimen (mm).

$$\sigma f = \frac{3E_t LP(E_c t_c^2 + 2E_c t_c t_t + E_t t_t^2)}{2w(E_c^2 t_c^4 + 4E_c E_t t_c^3 t_t + 6E_c E_t t_c^2 t_t^2 + 4E_c E_t t_c t_t^3 + E_t^2 t_t^4)} \qquad \text{Eq 2}$$

The flexural strength data for each design group were analyzed using one-way ANOVA ($\alpha = 0.05$), and Tukey post-hoc test ($\alpha = 0.05$) was used to identify differences among the cooling methods.

Results

The mean values (MPa), standard deviations, and coefficients of variance for three-point flexural strength are presented in Table 2. The results of one-way ANOVA for bilayer specimens 3Y-TZP/VM9 with porcelain on lower surface (PB) were statistically significant (Table 3). Tukey post-hoc test showed the highest flexural strength for fast cooling and the lowest for slow cooling; the normal cooling was not different from fast and slow cooling. The results of the one-way ANOVA for PM (Table 4), ZM (Table 5), and ZB (Table 6) were not statistically significant ($\alpha > 0.05$).

Table 2. Mean (MPa) of flexural strength, standard deviation, and coefficient of variance.

Group	Cooling	Average	Sd	Covar
PM	Slow	74.4	9.0	12.1%
	Normal	75.4	7.7	10.2%
	Fast	77.9	11.1	14.2%
ZM	Slow	835.5	129.9	15.5%
	Normal	873.0	140.1	16.1%
	Fast	817.1	95.2	11.6%
PB	Slow	49.3	10.1	20.5%
	Normal	56.1	11.0	19.6%
	Fast	67.5	16.7	24.8%
ZB	Slow	572.5	136.5	23.8%
	Normal	590.6	117.4	19.9%
	Fast	691.3	177.8	25.7%

Discussion

The null hypothesis was partially rejected. In monolayer specimens, the cooling method did not change the flexural strength. The lack of effect of the cooling rate for zirconia monolayer specimens was possibly because the temperature of simulating the feldspathic porcelain sintering was below the temperature required to induce phase transformation in the used Y-TZP. However, for porcelain monolayer specimens, could be expected thermal material tempering on fast cooling method due to very high heat transfer between the material and the environment[7,19-20] or microstructural changes on vitreous matrix on slow cooling[21-22]. Moreover, the PM group, as monolayer specimens, did not have any effect of thermal expansion coefficient mismatch on the framework material and consequent residual tension.

For bilayer specimens, in ZB groups, the cooling method was not able to affect the flexural strength because zirconia was on the lower surface and it was more directly under tension and might be responsible for the whole sample strength[23-24].

In PB groups, the cooling method affected the flexural strength. This could be attributed to residual tension. At temperatures above the glass transition temperature (Tg – around 600 °C) stresses are relieved by plastic deformation since the porcelain behaves as a viscoelastic liquid and allows the rearrangement of the atoms within the structure. When the temperature declines to the glass transition region, atomic displacement is more difficult to occur. Thus, the viscous liquid porcelain gets denser with the atoms in closer packing. At temperatures below the Tg, porcelain is solid and structural rearrangements are impossible. At this moment, residual stress develops from the potential discrepancy in volume, density and viscosity between layers of porcelain that are below (external) and above (internal) the glass transition phase. This process could be affected by the cooling rate, thickness, thermal conductivity, and the mismatch in coefficient of thermal expansion of both the porcelain and zirconia core[7-11].

In the fast cooling rate, the external regions cool faster

Table 3. One-way ANOVA results of flexural strength values of porcelain monolayer design

	Sum of squares	df	Mean Square	F-Ratio	Significance
Inter-group	97.58	2	48.792	0.5574	0.582
Intra-group	3680.00	42	87.527		
Total	3777.58	44			

Table 4. One-way ANOVA results of flexural strength values of zirconia monolayer design

	Sum of squares	df	Mean Square	F-Ratio	Significance
Inter-group	24,400.0	2	12,200.0	0.8025	0.541
Intra-group	638,000.0	42	15,200.0		
Total	662,400.0	44			

Table 5. One-way ANOVA results of flexural strength values of zirconia bilayer design

	Sum of squares	df	Mean Square	F-Ratio	Significance
Inter-group	123,000.0	2	61,500.0	2.8802	0.066
Intra-group	897,000.0	42	21,300.0		
Total	1020,000.0	44			

Table 6. One-way ANOVA results of flexural strength values of porcelain bilayer design

	Sum of squares	df	Mean Square	F-Ratio	Significance
Inter-group	2,530.0	2	1,270.0	7.5813	0.002
Intra-group	7,010.0	42	167.0		
Total	9,540.0	44			

and the temperature gradients through the porcelain increase, concentrating stresses near the surface. Thus, stress development increases with higher porcelain thickness, faster cooling rate, and lower thermal conductivity[10]. Moreover, the mismatch between thermal expansion coefficient and thermal gradients inevitably makes the layered structures subject to a high residual stress when cooled from a furnace temperature[11]. The highest flexural strength observed in fast cooling in the PB group might be associated to compressive forces on the surface. However, when those structures are subjected to cyclic loads such as chewing, chipping or delamination may occur. Thus, the dental laboratory technician must be careful with the fast cooling method.

The slow cooling method has been proposed to decrease the mismatch of coefficients of thermal expansion and thermal diffusion. It was expected that this method would allow cooling of both materials at a more uniform rate. However, annealing of the porcelain occurs when the restoration is cooling slowly. This reduces substantially the possibility of surface compressive force formation, which is believed to strengthen the restoration. However, this speculation was not confirmed in this study. In fact, the slow cooling method could be harmful since it adds more heat to the restoration and, thus, increases the potential for induction of thermal strains and possible zirconia phase transformation[25]. Nevertheless, the interaction of zirconia and porcelain during veneering requires more investigation, at several principles

of thermodynamics.

It is important to observe that flexural strength with bar samples is a simplified method for predicting clinical performance of these materials. However, fixed partial dentures may show a different behavior than bars due to their complex geometry. Additionally, in the oral environment these materials are susceptible to different chemical and physical fatigues that were not reproduced in this study.

Then, different cooling methods affected only the flexural strength of bilayer specimens with porcelain in lower surface. The complex residual thermal stresses generated in bilayer specimens could be associated with thermal tempering stress, which probably did not occur in the slow cooling method.

Acknowledgements

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