

Effect of the Cooling Rate on the Properties of Veneer Porcelain for Zirconia Dental Prosthesis

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Received: February 10, 2017; Revised: June 04, 2017; Accepted: July 16, 2017

To solve chipping of veneer porcelain in zirconia dental prosthesis it has been studied different cooling rates in firing schedules to control residual stresses generated by differences in thermal properties between the ceramics. However, cooling rates may affect the porcelain, irrespectively of interaction with zirconia. This study analyzed the effect of cooling rate on the three-point flexural strength (FS), residual stress, Vickers hardness (VH) and V-notch fracture toughness (SEVNB) on a feldspathic porcelain (Vita VM9) indicated for covering zirconia. Bar-shaped specimens were sintered according manufacturer's schedule firing and cooled at different rates (slow, normal or fast). Specimens were tested for FS (n=30), VH (n=15) and SEVNB tests (n=15). Weibull analysis were performed from FS values to measure reliability and probability of failure (PF). One-way ANOVA was significant for VH ($p < 0.001$), and Tukey's test showed that slow cooling led to higher values. There was no significant difference for FS ($p = 0.654$) and SEVNB ($p = 0.734$). Fast cooling led to the lowest PF, but cooling rate factor was not significant for Weibull modulus. All cooling group showed residual tensile stress (MPa): Slow = 34.81; Normal = 27.04; Fast = 18,98. It was concluded that cooling rate produce minor changes in the porcelain properties.

Keywords: *dental porcelain, cooling, material resistance.*

1. Introduction

Dental zirconia (Yttria Tetragonal Zirconia Polycrystal or Y-TZP) veneered with porcelain was introduced in the 2000's and have been widely used for single crowns and fixed partial dentures. Besides the suitable mechanical properties of zirconia¹, long-term clinical research has reported higher rates of veneer chipping (cohesive fracture of porcelain) compared to well-known metal-ceramic restorations¹⁻⁵. Chippings could be related to the residual stress⁶⁻¹⁶ generated by successive firings and cooling cycles during porcelain veneer sintering, as result of mismatch of thermal expansion coefficient (TEC) between porcelain veneer and zirconia, and differences in thermal diffusivity and thermal conductivity of the ceramic materials.

Firing schedule of porcelain reaches the maximum temperature between 750 °C and 1000 °C, when the ceramic becomes a plastic state. During cooling, as the temperature reaches the glass transition temperature (T_g), the ceramic changes from a plastic state to a solid state¹⁷. Below the T_g, the stresses generated due to differences between the TEC of zirconia and that of veneer cannot be relieved by plastic

flow because the porcelain's viscosity is highly increased¹⁷. Because of the high stiffness of ceramics materials, residual stresses are produced and it can increase the probability of porcelain veneer chipping during mastication^{18,19}.

Moreover, considering that zirconia-based substructures have lower thermal diffusivity and thermal conductivity if compared to metallic substructures for metal-ceramic restorations, several firing cycles with different cooling methods have been proposed, which vary antagonistically since slow cooling^{7,11,14,20,21} to fast cooling^{6,9,10,12,20-23}. However, there is no consensus about an ideal firing cycle. In addition, the manufacturer's information about cooling rate after sintering the porcelain is unclear. The manufacturer recommends opening partially the oven door (75% opening), cooling down until room temperature, but it is not informed a precise cooling rate.

While numerous studies have mostly been conducted on bilayer specimens (porcelain/zirconia), less attention has been paid to the possible effects of cooling methods on porcelain properties itself, irrespectively of the residual stresses generated by the bilayer porcelain/zirconia complex. Some studies have shown changes in the porcelain microstructure and TEC due successive firings and different sintering cycles

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design^{24,25}. Thus, it is speculated if more aggressive cooling rates proposed for zirconia/porcelain restorations might influence porcelain microstructure and properties.

The aim of this investigation was to compare the flexural strength, reliability and probability of failure by Weibull analysis, Vickers hardness, residual stress and V-notch fracture toughness of a dental feldspathic porcelain indicated for dental zirconia veneering following schedule firing with three different cooling rates. The null hypothesis was that there are no significant differences in flexural strength, Weibull modulus, Vickers hardness (VH) and V-notch fracture toughness (SEVNB) of a porcelain sintered according to different firing cycles.

2. Experimental

2.1 Preparation of specimens

The porcelain samples were made according to ISO 6872:2008²⁶. Vita VM9 ceramic powder (Vita Zahnfabrik, Bad Säckingen, Germany) and Vita Modeling Liquid (Vita Zahnfabrik) were mixed and inserted into a mold. Excess liquid was blotted with absorbent paper. The specimens were removed from the mold and placed on a vacuum sintering furnace (Aluminipress, EDG, Sao Carlos, SP, Brazil). The firing schedule was carried-out until reach the maximum temperature following the manufacturer's recommendations (Table 1), but for cooling to the room temperature, three different experimental cooling rate protocol were performed as follows:

Slow - the samples were left inside the closed, turned-off furnace until they reached ambient temperature (about 8 hours);

Normal -when the temperature inside the furnace reached 500 °C, the samples were removed and cooled at room temperature;

Fast - the samples were immediately removed from the furnace (at 910 °C) after the holding time and blasted by compressed air (less than 10 sec).

Table 1. Firing schedule for Vita VM9 Dentine porcelain

Pre-Drying time (min)	6
Pre-Drying Temperature (°C)	500
Heating Rate (°C/min)	55
Firing Temperature (°C)	910
Holding time (min)	1

2.2 Three-point flexural strength

Thirty bar-shaped specimens of each experimental protocols were ground using 120-, 220-, 320-, 400-, 600- and 1200-grit wet SiC paper discs (Norton Abrasivos, Sao Paulo, SP, Brazil) using a polishing machine (Metaserv 2000,

Buehler, Buehler UK Ltd., Coventry, United Kingdom), until final dimensions of 4 ± 0.25 mm in width, 1.2 ± 0.2 mm in thickness and 22 mm in length were obtained. The width and thickness dimensions of the specimens were measured with digital calipers (Mitutoyo Corporation, Tokyo, Japan) in 3 different locations (left, middle and right). The specimens were placed in a sample holder, which had a span of 15 mm between two 0.8 mm-radius rounded bearers, and were loaded until failure using 1.6 mm-radius, rounded, steel knife edges and a universal testing machine (DL 2000, EMIC, São José dos Pinhais, PR, Brazil), with a 5.0 kN load cell and at a crosshead speed of 1.0 mm/min. The test was performed in distilled water at 37 °C, with the load applied at the midpoints of the samples (n=30). The flexural strength was calculated according to equation 1:

$$\sigma_f = \frac{3FL}{2wh^2} \quad (1)$$

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

2.3 Weibull analysis

To assess the reliability of the porcelain with each cooling method, Weibull regression analysis was performed based on the flexural strength data to determine the Weibull modulus and characteristic strength (n=30), with a confidence interval of 95%. Equation 2 provides a description of the Weibull distribution:

$$P = 1 - \exp\left[\left(-\frac{\sigma}{\sigma_0}\right)^m\right] \quad (2)$$

where P is the probability of fracture, σ is the flexural strength, σ_0 is the characteristic strength at a fracture probability of 63.21%, and m is the Weibull modulus, which is the slope of the line plotted on the " $\ln[\ln [1/(1-P)]]$ vs $\ln \sigma$ " Cartesian plane.

2.4 Vickers hardness

Parts of the fractured flexural test specimens (n=15) were embedded in acrylic resin. These specimens were ground using sequential wet SiC paper discs with #4000-grit (Norton Abrasivos, Sao Paulo, SP, Brazil) in a polishing machine (Metaserv 2000, Buehler, Buehler UK Ltd., Coventry, United Kingdom). Nine Vickers indentations were created on each specimen using a microindentation tester (1600-6300 model, Buehler, Lake Bluff, IL, USA) with a peak load of 9.8 N and a dwell time of 20 s. The mean value was calculated.

2.5 Fracture toughness test

The fracture toughness was determined using the single-edge V-notched beam (SEVNB) method (n=10) according to ISO 6872:2008²⁶. After cooling, the specimens were rectified with a diamond wheel until to achieve final parallel dimensions of 4 mm x 3 mm x 22 mm. The specimens were notched using a razor blade and 3 μm diamond paste, with a machine specifically designed for this study to guide the razor blade linearly. The notches were cut across centrally on the 3 mm x 22 mm surfaces. The depths of the V notches (1.0 ± 0.2 mm) were measured using SEM micrographs obtained from the notch tip of each specimen. Figure 1 shows one of the samples with V notch to measure the K_{Ic} by SEVNB method. A four-point flexural test was performed until failure with the 3 mm-width face and with the V-notch down, using a universal testing machine (DL 2000, EMIC, São José dos Pinhais, PR, Brazil) in air with a 5.0 kN load cell and crosshead speed of 0.5 mm/min. The sample holder had a span between the two 0.8 mm-radius, rounded bearers of 16 mm. The distance between the two 0.8 mm-radius, rounded loading pistons was 8 mm.

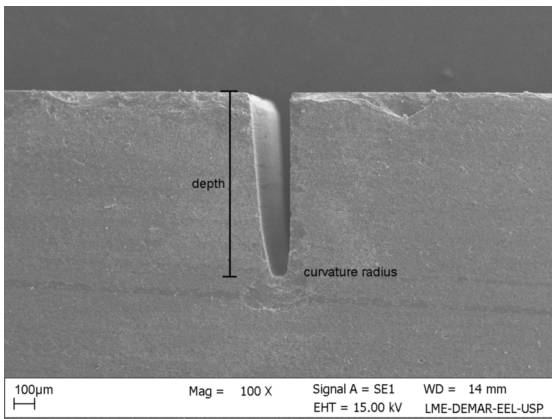


Figure 1. SEM micrograph and description of a V-notch.

Fracture toughness (K_{Ic}) was calculated based on the four-point flexural test results, using equation 3:

$$K_{Ic} = \frac{P}{b\sqrt{w}} \times \frac{S_1 - S_2}{w} \times \frac{3Y\sqrt{\frac{d}{w}}}{2\left(1 - \frac{d}{w}\right)^{\frac{3}{2}}} Y \quad (3)$$

where P is the maximum load (MPa), S_1 and S_2 are the outer and inner roller spans, respectively, b and w are the thickness and height of the specimen, respectively, d is the average depth of the notch, and Y is represented in equation 4:

$$Y = 1,9887 - 1,326 \frac{d}{w} - \frac{\left(3,49 - 0,68 \frac{d}{w} + 1,35 \left(\frac{d}{w}\right)^2\right) \frac{d}{w} \left(1 - \frac{d}{w}\right)}{\left(1 + \frac{d}{w}\right)^2} \quad (4)$$

2.6 Residual stress measurements

The fracture toughness was measured by indentation technique on porcelain surface before and after the slow, normal or fast cooling rate. Samples of VM9 of each group (n=5) was polished and the fracture toughness was measured by the Vickers hardness indentation technique. For each specimen three indentations (9,8N; 30s) were performed to calculate the fracture toughness (K_{Ic}). The specimens were submitted to the firing cycle followed by slow, normal and fast cooling. Then, a final fracture toughness was measured (K_{Ic}). Residual stress (MPa) was calculated according to the following equation¹³:

$$\sigma_r = \frac{K_{Ic} - K_{Ic'}}{2\sqrt{\frac{c}{\pi}}} \times 1000 \quad (5)$$

where, K_{Ic} is the fracture toughness measured before cooling, $K_{Ic'}$ is the fracture toughness measured after cooling (slow, normal or fast), and c is the half size of the median crack measured before the cooling.

2.7 Statistics

Statistical analyses were performed for the described tests using one-way analysis of variance ($\alpha < 0.05$), and by multiple comparisons were performed using Tukey's post hoc test.

2.8 Scanning electron microscope (SEM)

Three specimens of each group were examined in a scanning electron microscope (JEOL - JSM 6510, Jeol, Tokyo, Japan). Specimens were polished and etched with 2 % HF (hydrofluoric acid) for 15s and sputter-coated with gold-palladium. Three scanning electron micrographs from each group were obtained at a magnification of 500x.

3. Results and Discussion

The results of flexural strength (σ_f), Vickers hardness and fracture toughness are shown in Table 2.

The effect of thermal properties of zirconia and feldspathic porcelain on the chipping and delamination has been extensively studied^{16,7,9-12,14,20-23}, but it has not been considered the possible effect of the cooling rate exclusively on the porcelain. It is known that residual stresses can occur due to a thermal mismatch between the zirconia core and the porcelain veneer, or due to an inadequate cooling rate after the last firing of the porcelain veneer applied on zirconia^{11,12,17,23}. However, considering solely porcelain, stresses may also be generated due to a non-uniform solidification resultant from thermal gradients from the surface to the center of the porcelain^{7,12}, once the ceramic materials have low thermal diffusivity and low thermal conductivity. Tensile stress generated during

Table 2. Means of flexural strength, Vickers hardness and fracture toughness according to three different cooling methods.

Cooling	Flexural strength (MPa)	Vickers hardness (GPa)	Fracture toughness (MPa/m ^{1/2})
Slow	74.3 (8.27)A	5.41 (0.08)A	1.09 (0.13)A
Normal	75.1 (7.32)A	5.26 (0.10)B	1.02 (0.10)A
Fast	76.3 (9.58)A	5.21 (0.08)B	1.16 (0.18)A

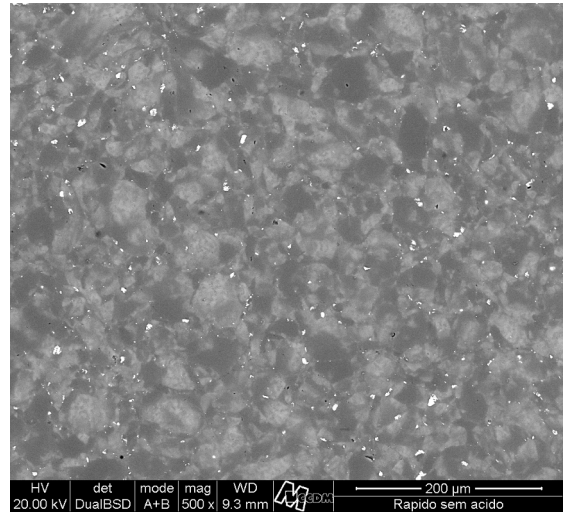
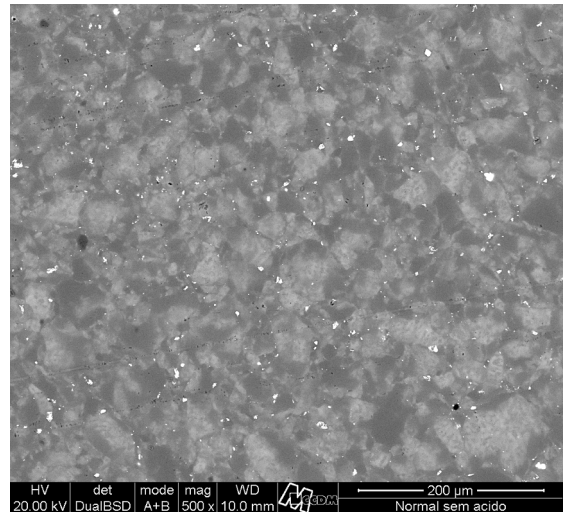
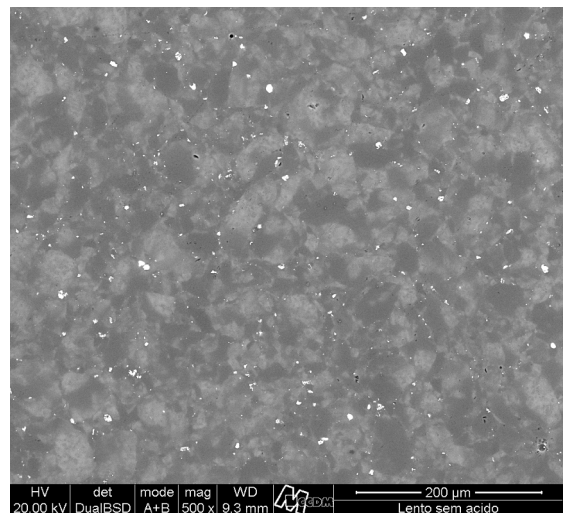
Same letters correspond to statistical similarity. Different letters correspond to statistical difference.

porcelain cooling is generally associated to veneer chipping and fracture¹³. In the present study, the residual stress test revealed that all the specimens showed tensile stress (MPa) at surface: Slow = 34.81; Normal = 27.04; Fast = 18,98. In this context, the slow cooling may make the porcelain more susceptible to failure.

The null hypothesis to three-point flexural strength and V-notch fracture toughness (SEVNB) were accepted. One-way ANOVA was not significant for flexural strength values ($F_{2,87}=0.436$, $\alpha=0.654$) or fracture toughness values ($F_{2,42}=3.220$, $\alpha=0.734$). These results are in agreement to the literature^{8,13,27}.

The three-point flexural strength test was performed in distilled water at 37 °C to simulate the oral environment, once the ceramics are constantly exposed to humid environment. Water can penetrate in a surface defect resulting in a rupture of the metallic oxides bonds, leading to the formation of hydroxyls²⁸. This phenomenon, known as slow crack growth (SCG), can control the time in service of the restoration²⁹. However, the short period to perform three-point flexural test may be insufficient to induce SCG, but the test becomes more challenging and the material more susceptible to fast crack growth. The mean value found to VM9 porcelain in normal cooling (75.1 ± 7.5 MPa) was different from those found by Fisher et al.⁸ (106.6 ± 12.5 MPa) who conducted the testing at dry environment. Borba et al.³⁰ performed the three-point flexural strength at 37 °C in artificial saliva and found characteristic strength (68.5 MPa) slightly lower than those found in our study to all cooling rates (slow: 78,03 MPa; normal: 78.62 MPa; Fast: 80.73 MPa). Thus, a possible explanation to the lower values compared to those found by Fisher et al.⁸ is the presence of water in the flexural strength test.

Morena et al.³¹ asserted that fracture toughness is principally dependent on the glass matrix. Nevertheless, some authors have pointed that porcelain with a higher content of leucite has higher fracture toughness^{20,27,32}. According to Mackert Jr. et al.³³, the quantity, average size and structure of the crystalline phase can directly affect the mechanical properties of the final product. On the other hand, Cesar et al.²⁸ compared porcelains with different leucite contents and found no direct relationship between the leucite content and slow crack growth. SEM images revealed comparable microstructures with crystal distribution similar among porcelain submitted to fast, normal or slow cooling (Figures 2, 3 and 4).

**Figure 2.** SEM images at 500 x magnification of Vita VM9 submitted to fast cooling.**Figure 3.** SEM images at 500 x magnification of Vita VM9 submitted to normal cooling.**Figure 4.** SEM images at 500 x magnification of Vita VM9 submitted to slow cooling.

The null hypothesis concerning to Vickers hardness (VH) was rejected, once one-way ANOVA was significant ($F_{2,39} = 3.238$, $\alpha < 0.001$). Tukey's test showed that slow cooling resulted in the highest hardness, while there was no difference between fast and normal cooling. The thermal history of porcelain may influence the content of leucite, such as the sintering time, the number of firings or the cooling rate^{24,34-36}. We speculated that slow cooling would allow additional crystal growth, and if it would justify the hardness improvement in slow cooling group. However, SEM images showed similar crystal distribution (Figure 2, 3 and 4), not giving evidence to support that statement. Although it was not possible to observe this phenomenon in SEM analysis, it would be interesting to perform other methodologies in another study to show the growth of crystals in the porcelain cooled slowly.

Weibull modulus and characteristic strength with a confidence interval of 95% is shown in Table 3). Higher Weibull modulus (m) was observed to normal cooling, but the confidence intervals showed no statistical difference to the porcelain cooling rates. According to Della Bona et al.³⁷, the reliability of structural ceramics is the most important factor in the clinical success of ceramic systems. High Weibull modulus values correspond to levels of high structural integrity and reliability. Most ceramics have values of " m " between 5 and 15, while " m " of metals vary between 30 and 100.

Table 3. Weibull modulus and characteristic strength with a confidence interval of 95% to the three different cooling methods.

Cooling	Characteristic strength (MPa)	Confidence Intervals	Weibull modulus	Confidence Intervals
Slow	78.03 ^a	74.65-81.46	9.83 ^a	6.88-12.63
Normal	78.62 ^a	75.53-81.74	10.86 ^a	7.60-13.96
Fast	80.73 ^a	76,74-84.80	8.59 ^a	6.01-11.04

Same letters correspond to statistical similarity. Different letters correspond to statistical difference.

According to Figure 5, probability of failure was higher for the normal and slow cooling samples. The probability of failure was calculated using a function of applied stress based on the Weibull probability plot. For example, at 90 MPa, all specimens of normal and slow cooling method failed, while approximately 10 % of fast cooling specimens were able to resist.

In a previous publication²¹, porcelain/zirconia bilayer specimens were used to investigate how the cooling rates influence the flexural and shear bond strength of feldspathic porcelain for zirconia. Using the same cooling method, it was observed that the fast-cooling rate showed a higher flexural strength but a lower reliability while the slow-cooling rate decreased the shear bond strength and showed lower adhesive failure mode. These results suggested that the cooling method may affect the longevity of zirconia-based

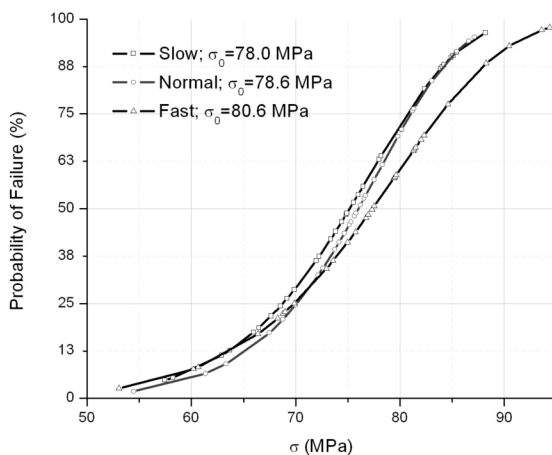


Figure 5. Probability of failure based on the flexural strength of the VM9 porcelain, according to cooling method.

restorations. In the present study, the cooling method did not influence the flexural strength results, which points out the role of the thermal properties of the framework in a bilayer complex. In a bilayer porcelain/zirconia, the compressive/tempering stresses in the porcelain layer under fast cooling can improve the resistance of the surface layer⁷ and develop high residual tension at the zirconia/porcelain interface^{6,9}. Thus, this phenomenon has great importance to bilayers. On the other hand, in this study, porcelain monolayer specimens could release partially the thermal stresses, once porcelain was not attached to zirconia.

With regard to the cooling rates, it is important to highlight the uncertainty about the best thermal cycling completion. Nowadays, VITA VM9 manufacturers recommends "Long-term cooling down to the respective temperature is recommended for the respective last firing cycle of the veneering ceramic; the lift position for VITA VACUMAT furnaces should be > 75%. Firing objects must be protected against direct supply of air". However, this information is unclear, not showing a precise cooling rate. Furthermore, there is no standard for cooling nomenclature²¹. In this study, the "normal" cooling technique was an approach to that recommended by the manufacturer, once our proposal was to test the effect of extreme cooling rates (slow and fast) to clarify their influence on selected mechanical properties of the porcelain.

It must be highlighted some limitations of this study. Results obtained from bar specimens may not be extrapolated to complex shapes as dental crowns. In addition, it was not identified and quantified crystalline phase resultant from each cooling protocol.

4. Conclusions

It was concluded that cooling rate produce minor changes in the porcelain properties when porcelain is considered in isolation, that is, unattached to a zirconia base, once flexural strength, fracture toughness and Weibull modulus were not affected by cooling method, despite fast cooling

had lowered probability of failure and slow cooling had improved Vickers hardness.

5. Acknowledgements

This research was supported by research grant FAPESP (2009/17735-8 and 2010/05469-9).

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