

The effect of prolonged holding time in firing schedules on the bond strength between the zirconia core and veneered porcelain

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Purpose: The purpose of the present study was to determine the effect of prolonged holding time in firing schedule to the bonding strength of zirconia core to the veneered porcelain.

Materials and Methods: Twenty cubes of zirconia (Y-TZP, Cercon Base 47) were prepared and veneering ceramics (Vintage ZR) were layered. The bond strengths of zirconia core and layered porcelain with different holding time in firing schedule were evaluated by shearing test.

Results: The shear bond strengths of zirconia core and veneered porcelain with different holding time showed a statistically significant difference, 38.8±5.6 MPa (holding time; 1 minute) and 44.0±4.2 MPa (holding time; 21 minutes).

Conclusion: Prolonged holding time in firing schedule will improve the bond strength of zirconia core and veneered porcelain which reduce chipping failure of zirconia-based all ceramic restorations.

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Key Words: chipping, firing schedule, shear bond strength, thermocouple, zirconia ceramics

Introduction

Metal-ceramic restorations have been the most popular esthetical restoration method in prosthodontic field. This restoration used to be a completed method in terms of strength and fracture resistance.¹ But restorations with metal revealed to have some problems such as esthetical problem, metal allergy problem. For these reasons, restorations without metal (metal free restorations) have made an appearance in this field. Among them, all ceramic restoration systems with zirconia core have been using for its superior mechanical properties such as high flexural strength, high rigidity, and good biocompatibility.^{2,3} Fine ceramics such as alumina and zirconia have been used in industrial field for their excellent mechanical properties. But because of their toughness, it was not easy to apply them in dental field.

The introduction of CAD/CAM technology into the dental field has facilitated the use of zirconia; yttria-partially stabilized zirconia (Y-TZP). As the zirconia restoration has become widely used, many clinical reports pointed out the clinical failure of chipping of layered porcelain on crown and bridges with zirconia frame.⁴⁻⁷ Some studies reported that the bond strength of zirconia to veneering porcelain is not strong enough.⁸⁻¹⁰ Fracture patterns observed in failed crowns were cohesive failure within the veneering ceramic or adhesive failure at the core-veneer interface.¹¹ But some reported that a significant incidence of cohesive fracture in the veneering porcelain has been observed.^{12,13} Guazzato et al.¹⁴ reported that the fracture mechanism of the porcelain layer (cohesive rather than adhesive) indicates that such as the design of the zirconia frameworks, the mechanical properties of the veneering porcelain or a mismatch of the coefficient of thermal expansion between porcelain and zirconia have been implicated.

The bond strength between zirconia frame and veneered porcelain will be influenced by various factors, such as the firing process of veneered porcelain, surface treatments of the frame. As for the firing process, mismatch

in the heat expansion coefficients of zirconia and porcelain will cause the residual stress¹⁴⁻¹⁶ and low thermal conductivity of zirconia will cause incomplete baking of porcelain. Both of them will lead to the clinical failure of chipping. As for the surface treatment of the frame, sandblasting, laser, and zirconia powder coating have been applied to make the surface of zirconia frame coarse.¹⁷⁻¹⁹ For zirconia is a chemically inert matter, it cannot be etched by HF or H₃PO₄. But any surface adjustment, such as grinding, sandblasting, and even polishing can change the phase on the surface of the zirconia which may affect the stability and strength of the zirconia as well as the veneer porcelain.

During the heating and cooling process, difference of thermal conductivity between zirconia and layered porcelain will cause residual stress which may lead to the delamination of layered porcelain.¹⁶ Therefore, many reports on the effect of changing firing schedule on the bond strength between zirconia core and veneered porcelains.¹⁴⁻²⁶

In this study, we focused on the low thermal conductivity of zirconia. Because of the low coefficient of heat conduction of zirconia, surface temperature of zirconia framework will not be increased to gain enough bonding strength between zirconia core and veneered porcelain during firing process. And this can be avoided by the prolonged holding time. There are few reports focused on this point. Therefore, the purpose of this study was to determine the effect of prolonged firing time to the bonding strength of zirconia frameworks to the veneered porcelain. The null hypotheses of this study were that: prolonged firing time does not improve the bond quality of zirconia framework to veneering porcelain.

Materials and Methods

Experiment 1 Thermocouple measurement of the furnace temperature and temperature of the zirconia specimens' surface during firing schedule

To investigate the influence of low coefficient of heat conduction of zirconia on the aspect of zirconia frame surface temperature during firing process, Type-R (Pt-Rd) thermocouple device (Type-R thermocouple, Omega Engineering Inc, Stamford, CT, USA) was used to measure the surface temperature of Y-TZP specimen and furnace temperature. A cubical shaped full-sintered zirconia block (8.0x8.0x8.0 mm) with conical frustum-shaped hole to insert a thermocouple probe was prepared (Fig. 1). This block was made from the semi-sintered zirconia which was sintered in a furnace (Cercon Heat, Degudent GmbH, Hanau-Wolfgang, Germany) for 6.5 hours at 1,350°C. Porcelain furnace (TD-PF1, Tokuyama Dental Corp, Tokyo, Japan) was used in this study. Type-R thermocouple probe was set in the furnace to measure the temperatures of zirconia specimens in the furnace. The zirconia specimens were set on a block made with a phosphate-bonded investment material (Velvety, Shofu Inc, Kyoto, Japan) and covered the upper side with 8 mm thickness of Ceramic Putty (Thermeez, Cotronics Corp, Brooklyn, NY, USA) which was used as a heat insulating material. The schematic explanation of this setting was shown in Fig. 2.

According to the firing schedule of porcelain which was recommended by manufacturer, firing schedule in this experiment was set as follows: pre-drying temperature, 650°C; drying time, 6 minutes; final temperature, 920°C; heating rate, 45°C/minute; and holding time, 30 minutes. The temperature in the furnace was read by the indicated values of the display in the front panel of the furnace. And the temperature of zirconia specimens' surface was read by the value of thermocouple and digital thermometer (53 Series II Thermometer, Fluke Corp, Everett, WA, USA) (Fig. 3). Measuring results were simultaneously recorded by a digital video camera

(Handycam, SONY Corp, Tokyo, Japan). And the data (the values of temperature of the furnace and zirconia surface) were input into computer to plot the change of the temperature of zirconia and furnace against time. Time lag between the time when the furnace temperature was reached to the firing temperature and the time when the zirconia surface temperature was reached to the firing temperature were analyzed. The temperature of the zirconia surface was also measured at the time when the furnace temperature reached to the firing temperature.

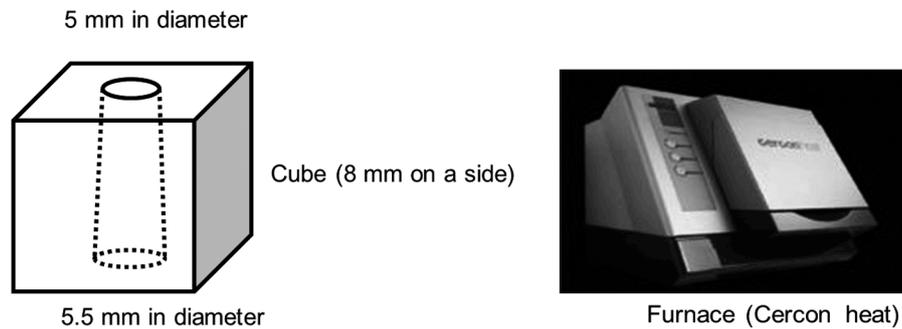


Fig. 1. Shape of test specimen and furnace for sintering

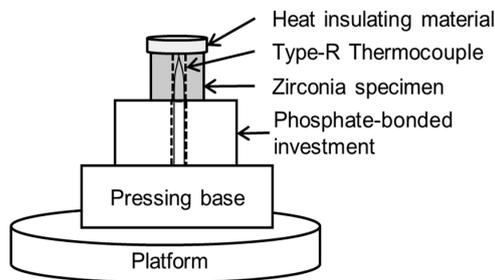
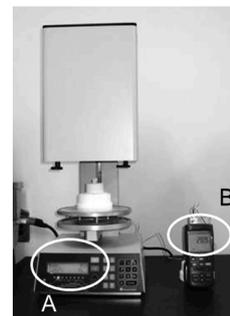


Fig. 2. Scheme of setting of specimen



A:Temperature in the furnace
B:Digital temperature indicator (Thermocouples)

Fig. 3. Temperature measuring scenario

Experiment 2 Shear test for bonding strength between zirconia core and veneered porcelain

To evaluate the bonding strength of zirconia core and layered porcelain obtained by different holding time during firing, we performed following experiment. Twenty cubes of Y-TZP (Cercon Base 47, Degudent GmbH, Rodenbacher, Germany) were prepared with an edge length of 8 mm. One face of each cube was polished with abrasive discs (CarbiMet II #300, #600, #800, and #1,200, Buehler, Lake Bluff, IL, USA) on a polisher (Ecomet, Buehler). After polishing, airborne particle abrasion (Hi-Blaster III, Shofu Inc) was performed on the polished face with 70-µm alumina (Hi Aluminas, Shofu Inc) for 20 s at a pressure of 0.2 MPa and with a distance of 10 mm between the nozzle and the surface of the cube. The cubes were cleaned with distilled water for 10 min. in an ultrasonic bath (SUC-35, Shofu Inc).

On the prepared face of each cube, two layers of veneering ceramic (cervical and body, Vintage ZR, Shofu Inc) which was recommended for veneering zirconia core, was added. First layer (cervical porcelain) was added covering an area of 2.5 mm diameter and 250 µm thickness on the prepared face, second layer (body porcelain) was added (2.5 mm diameter and 2 mm thickness) on the first layer which was fired by the firing schedule using a separable silicone mold made with an addition cure-type silicone impression material for laboratory use

(Duplicone, Shofu Inc). Firing cycles for veneering porcelain was shown in Table 1.

Table 1. Firing cycles for veneering porcelain according to manufacturer's instructions

Step	Atmosphere	Standby temperature (°C)	Drying time (minute)	Rate of temperature increase (°C/minute)	Final temperature (°C)	Holding time (minute:s)
Cervical porcelain	Vacuum	650	6	45	920	1:00
Body and Enamel	Vacuum	650	6	45	920	1:00
Glazing	Air	650	6	45	920	0:30

Test piece were fabricated under two experimental conditions (holding time; 1 minute and 21 minutes) for 10 pieces each. The completed specimen was inverted on a brass slab with a 4 mm diameter hole. Then an aluminum ring (20 mm in diameter and 30 mm in height) was placed over the specimens. Autopolymerizing resin (Palapress Vario, Heraeus Kulzer GmbH, Hanau, Germany) was mixed and poured into the ring. This mounting procedure was illustrated in Fig. 4. For shear bond test, each specimen was mounted in a metal holder on the universal testing machine (Autograph AGS-H, Shimadzu Corp, Kyoto, Japan) and the load was applied with the compression shear test jig (Custom-made) that had a chisel-shaped edge (0.5 mm of front edge thickness) (Fig. 5). Each specimen was tightened and stabilized to ensure that the edge of the shearing jig was touching the core surface and was positioned as close to the veneer-core interface as possible.

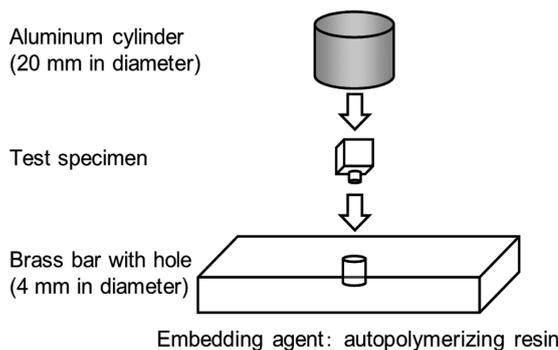


Fig. 4. mounting technique

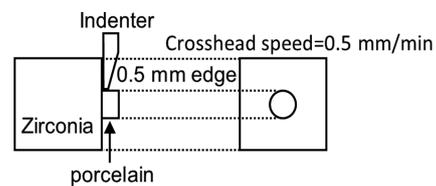


Fig. 5. Schema of shear test

Statistical analysis

To determine the effects of prolonged holding time on the shear bond strength, Student t-test was used after Levene's test for equality of variance. The test was performed at a level of significance of 0.05. The analyses were performed with the software for statistical analysis (SPSS 12.0J for Windows, SPSS Inc, Chicago, IL, USA).

Results

Experiment 1

The time differences were calculated from the time of zirconia to reach the target firing temperature and the time of furnace to reach the firing temperature (Fig. 6). The lag between the time for the temperature of zirconia

specimen and the furnace temperature arrived to the target firing temperature were as follows, 17 minutes 55 s, 19 minutes 15 s, 20 minutes 10 s, and mean, 19 minutes 7 s. The temperature of zirconia specimens were lower ($89.8 \pm 6.1^\circ\text{C}$) than the furnace temperature at the time when the furnace temperature reached to the firing temperature.

Experiment 2

The shear bond strengths were 38.8 ± 5.6 MPa (holding time; 1 minute) and 44.0 ± 4.2 MPa (holding time; 21 minutes) respectively which had a statistically-significant difference (Fig. 7).

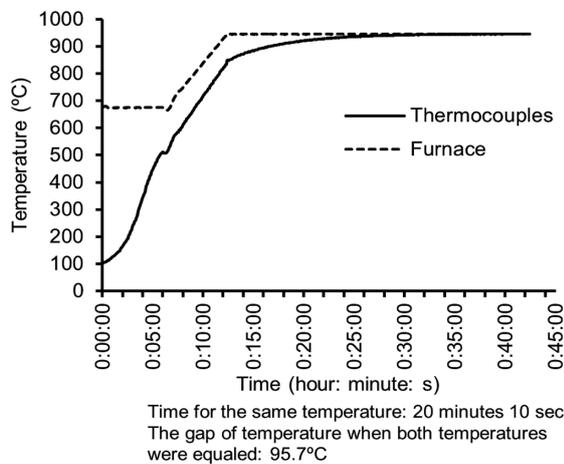


Fig. 6. Temperature on zirconia surface and inside of the furnace

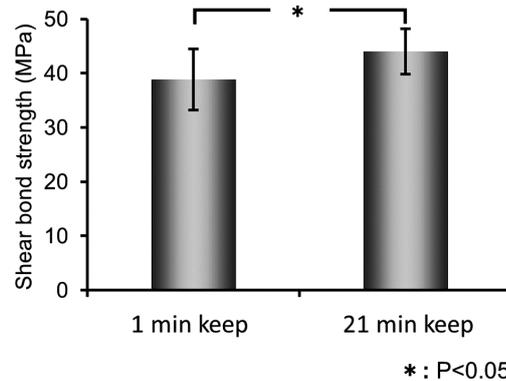


Fig. 7. Shear bond strength

Discussion

For measuring the temperature of zirconia specimens' surface in the furnace, thermocouple probe was set on the surface of zirconia specimen and covered with an insulating material to block out the effect of furnace temperature. Coefficient of thermal conductivity of the insulating material used in this study (0.16 W/mK) was lower than that of zirconia (2.7 W/mK). This means that temperature rise speed of the insulating material was slower than that of zirconia. For this reason, this method was suitable to block out everything but the temperature of the zirconia specimen. The zirconia specimen cubes with an edge length of 8 mm were designed to assume a zirconia pontic form for missing posterior teeth. The bulk of pontic will make the temperature rising of zirconia pontic part slower than the abutment part during the firing process of porcelain in the furnace. The result in this study shows that the lag between the time for the temperature of zirconia specimen and the furnace temperature arrived to the target firing temperature was 19 minutes 7 s on average.

Furthermore, the temperature of zirconia specimens were lower ($89.8 \pm 6.1^\circ\text{C}$) than the furnace temperature at the time when the furnace temperature reached to the firing temperature. These results indicate that the holding time (usually 1 minute) recommended by manufacturer is not enough for zirconia pontic to fuse porcelain completely in firing process. The bond strength between zirconia frame and veneered porcelain will be influenced by various factors, such as the firing process of veneered porcelain, surface treatment of frame, the design of zirconia frame. As for the firing process of veneered porcelain, four ways can be cited as possible solutions of this problem as follows; 1) change of cooling rate, 2) change of firing cycles, 3) rise of firing temperature, and 4) extension of the holding time.

For the solution 1, there are many reports.^{14,15,24} Many of them concluded that the fast cooling generated

significant residual stress within the veneering porcelain and slow cooling is effective to prevent clinical chipping failures. For the solution 2, Queiroz et al. reported that the number of porcelain firing cycles significantly affected the bond strength of the ceramic system.²⁵ For the solution 3, Hata et al. reported on the influence of firing temperature on the bond strength between zirconia core and veneering porcelains. The micro-tensile bond strength was significantly smaller when fired at a temperature 30°C below the recommended temperature but no significant difference under the condition at a temperature 30°C above the recommended temperature.²² In this study we examined the validity of the solution 4. Zirconia is a thermal insulator. This property prevents heat transfer to the veneer porcelain and thus prevents it from becoming fully dense if fired fast and at a low temperature. Fast cooling creates stress in the porcelain that can lead to cracking. Slow cooling may prevent stress from building on the surface of the porcelain, if it is not fully dense to start, slow cooling will fix the problem of insufficient density. Holding times were set (Table 1) on the basis of the result of experiment 1: Condition 1, 1 minute which is recommended by manufacturers; and Condition 2, 21 minutes, 20 minutes which needs for zirconia core temperature to arrive the target firing temperature plus 1 minute (recommended time).

For evaluating the bond strength between zirconia frame and veneered porcelain, various kinds of tests have been employed, such as the shear bond strength test,²⁷ three and four point loading test,^{28,29} and the microtensile bond strength test.^{30,31} In this study, shear bond strength test was selected because of its relatively simple procedure. Furthermore, clinical failure of chipping often caused in the area without the support of zirconia-frame and this indicates the process of chipping maybe caused by shear stress.

The shear bond strengths were 38.8±5.6 MPa (holding time; 1 minute) and 44.0±4.2 MPa (holding time; 21 minutes) respectively which had a statistically-significant difference. This result indicated that the extension of the holding time could make the temperature of the zirconia-porcelain interface reached to the target firing temperature, which made the firing process entirely completed. Because of the low coefficient of heat conduction of zirconia, surface temperature of zirconia framework will not be increased to gain enough bonding strength between zirconia core and veneered porcelain during firing process of veneered porcelain. And this can be avoided by the extension of the holding time. If the firing process were not entirely completed, mechanical properties of porcelain will not be enough and it will coincide the report¹⁴ that fracture mechanism of the porcelain layer (cohesive rather than adhesive) indicates the mechanical properties of the veneering porcelain play a significant role. In this study we set the holding time at 21 minutes, which is rather long, so we need further study to decide the proper holding time to get enough bond strength between the zirconia core and veneered porcelain. In conclusion, prolonged holding time for firing schedule will improve the bond strength of Y-TZP and layered porcelain which reduce chipping failure of zirconia-based all ceramic restorations.

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References

1. Fischer H, Marx R. Fracture toughness of dental ceramics: comparison of bending and indentation method. *Dent Mater* 2002; 18: 12-9.
2. Denry I, Kelly R. State of the art of zirconia for dental applications. *Dent Mater* 2008; 24: 299-307.
3. Kelly R, Denry I. Stabilized zirconia as a structural ceramic: an overview. *Dent Mater* 2008; 24: 289-98.
4. Vult von Steyern P, Carlson P, Nilner K. All-ceramic fixed partial dentures designed according to the DC-Zircon technique. A 2-year clinical study. *J Oral Rehabil* 2005; 32: 180-7.

5. Sailer I, Fehér A, Filser F, Lüthy H, Gauckler LJ, Schärer P, et al. Five-year clinical results of zirconia frameworks for posterior fixed partial dentures. *Int J Prosthodont* 2007; 20: 383-8.
6. Aboushelib MN, Feilzer AJ, Kleverlaan CJ. Bridging the gap between clinical failure and laboratory fracture strength tests using a fractographic approach. *Dent Mater* 2009; 25: 383-91.
7. Nozaki K, Kawazu A, Kizuki Y, Ueda Y, Omori S, Nemoto R, et al. Clinical evaluation of all ceramic fixed partial dentures fabricated with zirconium oxide. *Annal Jpn Prosthodont Soc* 2010; 2(119th Special Issue): 88.
8. Aboushelib MN, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different component of core veneered all-ceramic restorations. Part 3: double veneer technique. *J Prosthodont* 2008; 17: 9-13.
9. Choi BK, Han JS, Yang JH, Lee JB, Kim SH. Shear bond strength of veneering porcelain to zirconia and metal cores. *J Adv Prosthodont* 2009; 1: 129-35.
10. Heintze SD, Rousson V. Survival of zirconia-and metal-supported fixed dental prostheses: A systematic review. *Int J Prosthodont* 2010; 23: 493-502.
11. Aboushelib MN, Feilzer AJ, Kleverlaan CJ. Bridging the gap between clinical failure and laboratory fracture strength tests using a fractographic approach. *Dent Mater* 2009; 25: 383-91.
12. Raigrodski AJ, Chiche GJ, Potiket N, Hochstedler JL, Mohamed SE, Billiot S, et al. The efficacy of posterior three-unit zirconium-oxide-based ceramic fixed partial dental prostheses: a prospective clinical pilot study. *J Prosthet Dent* 2006; 96: 237-44.
13. Sailer I, Fehér A, Filser F, Gauckler LJ, Lüthy H, Hämmerle CH. Five-year clinical results of zirconia frameworks for posterior fixed partial dentures. *Int J Prosthodont* 2007; 20: 383-8.
14. Guazzato M, Walton TR, Franklin W, Davis G, Bohl C, Klineberg I. Influence of thickness and cooling rate on development of spontaneous cracks in porcelain/zirconia structures. *Aust Dent J* 2010; 55: 306-10.
15. Swain MV. Unstable cracking (chipping) of veneering porcelain on all-ceramic dental crowns and fixed partial dentures. *Acta Biomater* 2009; 5: 1668-77.
16. Ozkurt Z, Kazazoglu E, Unal A. In vitro evaluation of shear bond strength of veneering ceramics to zirconia. *Dent Mater J* 2010; 29: 138-46.
17. Kim HJ, Lim HP, Park YJ, Vang MS. Effect of zirconia surface treatments on the shear bond strength of veneering ceramic. *J Prosthet Dent* 2011; 105: 315-22.
18. Teng J, Wang H, Liao Y, Liang X. Evaluation of a conditioning method to improve core-veneer bond strength of zirconia restorations. *J Prosthet Dent* 2012; 107: 380-7.
19. Liu D, Matinlinna JP, Tsoi JK, Pow EH, Miyazaki T, Shibata Y, et al. A new modified laser pretreatment for porcelain zirconia bonding. *Dent Mater* 2013; 29: 559-65.
20. Fischer J, Stawarczyk B, Trottmann A, Hämmerle CH. Impact of thermal misfit on shear strength of veneering ceramic/zirconia composites. *Dent Mater* 2009; 25: 419-23.
21. Göstemeyer G, Jendras M, Dittmer MP, Bach FW, Stiesch M, Kohorst P. Influence of cooling rate on zirconia/veneer interfacial adhesion. *Acta Biomater* 2010; 6: 4532-8.
22. Hata U, Uehara Y, Sakurai Y, Yamamura O, Fujiwara S, Doi Y. Influence of firing temperature on the bond strength between zirconia core and veneering porcelains. *J J Dent Mater* 2010; 29: 112.
23. Rues S, Kröger E, Müller D, Schmitter M. Effect of firing protocols on cohesive failure of all-ceramic crowns. *J Dent* 2010; 38: 987-94.
24. Choi JE, Waddell JN, Swain MV. Pressed ceramics onto zirconia. Part 2: Indentation fracture and influence of cooling rate on residual stresses. *Dent Mater* 2011; 27: 1111-8.
25. Queiroz JR, Benetti P, Massi M, Junior LN, Della Bona A. Effect of multiple firing and silica deposition on the zirconia-porcelain interfacial bond strength. *Dent Mater* 2012; 28: 763-8.
26. Zeighami S, Mahgoli H, Farid F, Azari A. The effect of multiple firings on microtensile bond strength of core-veneer zirconia-based all-ceramic restorations. *J Prosthodont* 2013; 22: 49-53.
27. Guess PC, Kulis A, Witkowski S, Wolkewitz M, Zhang Y, Strub JR. Shear bond strengths between different zirconia cores and veneering ceramics and their susceptibility to thermocycling. *Dent Mater* 2008; 24: 1556-67.
28. White SN, Miklus VG, McLaren EA, Lang LA, Caputo AA. Flexural strength of a layered zirconia and porcelain dental all-ceramic system. *J Prosthet Dent* 2005; 94: 125-31.
29. Tan JP, Sederstrom D, Polansky JR, McLaren EA, White SN. The use of slow heating and slow cooling regimens to strengthen porcelain fused to zirconia. *J Prosthet Dent* 2012; 107: 163-9.
30. Aboushelib MN, de Jager N, Kleverlaan CJ, Feilzer AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. *Dent Mater* 2005; 21: 984-91.
31. Aboushelib MN, Kleverlaan CJ, Feiler AJ. Microtensile bond strength of different components of core veneered all-ceramic restorations. Part II: zirconia veneering ceramics. *Dent Mater* 2006; 22: 857-63.

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