# Thermal Tempering Effects on Flexural Strength of CAD-on Veneering Zirconia Restorations

# ผลของความเร็วในการลดความร้อนหลังการเผาต่อความทนแรงดัดของวัสดุบูรณะฟันเซอร์โคเนียที่เคลือบผิวด้วยแคดแคมเซรามิค

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**ABSTRACT**

The purpose of this study was to assess the influence of different cooling rate on the flexural strength of CAD-on veneering zirconia. Forty-two bars of IPS e.max ZirCAD (4*0.7*25 $mm^3$) were veneered with 1 mm. thickness of IPS e.max CAD using three different cooling regimens (TºC muffle open after firing) : fast (600 ºC), medium (Glass transition temperature), and slow(200ºC) cooling. All specimens were subjected to four point bending test. Flexural stress were calculated using mechanic and composite beam theory and the reliability was analyzed with Weibull distribution. One-Way ANOVA statistic analysis indicated that there was no significant difference in mean flexural stress among this three groups(P>0.05). It can be concluded that different cooling rate may not affect the flexural strength when using CAD-on veneering technique.

**บทคัดย่อ**

การศึกษานี้มีวัตถุประสงค์เพื่อศึกษาผลของความเร็วในการลดความร้อนหลังการเผาต่อความทนแรงดัดของวัสดุบูรณะฟันเซอร์โคเนียที่เคลือบผิวด้วยแคดแคมเซรามิค โดยนำแท่งไอพีเอส อีแมกซ์เซอร์แคดขนาด 4*0.7*25 ม.ม.$^3$ ทั้งหมดจำนวน 42 ชิ้นมาเคลือบผิวด้วยไอพีเอส อีแมกซ์แคดหนา 1 ม.ม. ซึ่งจะแบ่งเป็น 3 กลุ่มตามอุณหภูมิเปิดเตาได้แก่ลดความร้อนเร็ว(600 องศาเซลเซียส)ลดความร้อนเร็วปานกลาง (อุณหภูมิกลาสทรานซิชั่น)และลดความร้อนช้า (200องศาเซลเซียส) ชิ้นงานทุกชิ้นจะถูกทดสอบด้วยวิธีการทดสอบแบบกด 4 จุดทำการวิเคราะห์ค่าความทนแรงดัดด้วยทฤษฎีเชิงกลและทฤษฎีคอมโพสิตบีม และวิเคราะห์ความเชื่อถือได้จากการวิเคราะห์การกระจายตัวของความแข็งแรงใช้สถิติการวิเคราะห์ความแปรปรวนแบบทางเดียวในการวิเคราะห์ข้อมูล กำหนดนัยสำคัญที่ระดับ 0.05 การศึกษานี้พบว่า มีความแตกต่างอย่างไม่มีนัยสำคัญทางสถิติของค่าเฉลี่ยความทนแรงดัดระหว่างกลุ่มทดลอง 3 กลุ่มนี้ จึงสรุปว่าความเร็วในการลดความร้อนหลังการเผาอาจไม่มีผลต่อความทนแรงดัดเมื่อเคลือบผิววัสดุบูรณะฟันเซอร์โคเนียด้วยวิธีแคดออน

**Key Words:** Zirconia base restoration, CAD/CAM, Flexural strength

**คำสำคัญ:** วัสดุบูรณะฟันเซอร์โคเนีย แคดแคม ความทนแรงดัด

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**Introduction**

Nowadays, Yttrium stabilized zirconia restoration has been widely used due to their high flexural strength and fracture toughness, as well as the achievement in esthetic outcome compared to the porcelain fused to metal restoration (Rues S et al., 2010). Nevertheless, Minor chipping of veneering porcelain was described as the most frequent failure of zirconia based restoration (Komine et al., 2010; Schmitter et al., 2012). In general, the veneering techniques have been used according to the well-known fabrication process of the metal ceramic technique. A minimum flexural strength of 50MPa for veneering glass ceramics is required according to ISO 6872 and 9693 standards (Beuer et al., 2009). Recently, a new technique using a CAD/CAM-fabricated high-strength zirconia coping and a corresponding CAD/CAM fabricated lithium-disilicate glass-ceramic veneering material has been reported (Schmitter et al., 2012; Beuer et al., 2009; Choi YS et al., 2012). Lithium-disilicate reinforced glass-ceramics exhibiting a flexural strength of 350-400MPa (Beuer et al., 2009; Choi YS et al., 2012; Guess et al., 2011). It is considerably stronger than conventional veneering ceramic. The advantages of these technologies are the reliability of restorations from decreasing in the pore and impurity that is a starting point for cracking and fracture in ceramic restoration. In other words, using the same ceramic material in the form of an industrial prefabricated block and applying the milling technique has higher Weibull modulus than other fabricated techniques (Beuer et al., 2009; Choi YS et al., 2012).

However, the durability of veneered zirconia core restoration is not only limited by the strength of the material used for fabricating the restoration, but also affected by the veneering process. Aboushelib et al. (2009) reported that fracture patterns of clinical failure can occur, and it can be either cohesive failure (chipping) or adhesive failure at the core veneer interface (delamination). One possible explanation that relates to this clinical complication is the extensive residual thermal stresses between the core and veneer developed during the cooling of the restoration from the sintering temperature to room temperature. Residual thermal stresses can be generated by the coefficient of the thermal expansion mismatch between the core and veneer and by a thermal gradient throughout the veneer layer at the temperatures that the veneering ceramic transforms from a viscoelastic into a solid state (Göstemeyer et al., 2010). Therefore, the cooling rate in the porcelain firing processes is one of the factors that affect the reliability of zirconia based restorations (Tan et al., 2012).

There is little information available about the behavior of CAD-on veneering zirconia restoration as well as the effects of the cooling rate on the strength of this veneering technique.

**Objective of the study**

The aim of this study was to assess the influence of different cooling rates on the flexural strength of CAD-on veneering zirconia using the four point bending test.

**Materials and methods**

**Core specimen preparation**

Forty-two bars of Yttrium-stabilized zirconia (Y-TZP, IPS e.max ZirCAD, Ivoclar Vivadent, Schaan, FL) were prepared from the partial sintered block using a diamond coated wheel

(Isomet® 1000 Precision, Buehler, Illinois, USA) into rectangular beam and ground down with a silicon carbide abrasive paper until 600 grit in order to reach the dimension 5 mm (width) × 31.25 mm. (length) × 0.9 mm. (height). All zirconia beams were cleaned under running water and dried in a drying cabinet. Then, sintered in the furnace (inFire HTC speed, Sirona Dental systems, Bensheim, Germany) at 1500ºC, holding time 4 hrs (5ºC/min both heating and cooling rates).The sintered zirconia core had a reduced volume of approximately 20%. The final dimension of each bar was 4 mm (width) × 25 mm. (length) × 0.7 mm. (height). The dimensional error in each specimen was accepted at ± 0.01 mm.

**CAD-on veneering technique**

Forty-two pre-crystalline lithium-disilicate glass ceramic bars were cut from IPS e.max CAD HT block (Ivoclar Vivadent, Schaan, Lichtenstein) using a diamond coated wheel (Isomet® 1000 Precision, Buehler, Illinois, USA)and ground down with a silicon carbide abrasive paper 150 grit in order to reach the dimension 5 mm (width) × 27 mm. (length) × 1.1 mm. (height).Forty-two beams of sintered zirconia core and veneering cap were then randomly divided into three groups in order to be veneered according to the three different cooling rates. For the veneering technique, the two components of zirconia bar and veneering cap were joined together using IPS e.max CAD Crystall/ Connect (Ivoclar Vivadent, Schaan, Lichtenstein). Excess material was evenly squeezed out and removed with a brush. Three of each of the specimens were arranged in a fibrous pad firing supports to ensure a homogeneous heat distribution. The specimens were fired at 840ºC according to the manufacturer's instruction except the cooling rate that was set to fast cooling, medium cooling, and slow cooling. For the fast cooling group, the furnace was set according to the manufacturer's instruction (fully open after the muffle temperature cooling down to 600 ºC). Second, the medium cooling group, the muffle was fully opened after the muffle temperature cooled down to 560ºC (glass transition temperature, Tg). Lastly, the slow cooling group, specimens were allowed to be left in the muffle until the muffle temperature cooled down to 200 ºC and then the muffle was fully opened. All specimens were removed from the muffle when the ambient temperature was reached.

After the sintering process, all specimens were adjusted and finished on the veneered surface using silicon carbide abrasive paper from 600 to2000 grit in order to reduce flaws and ensure the uniform thickness of veneering porcelain at 1.0 mm.. The total dimension of each specimen was 4 mm (width) × 25 mm. (length) × 1.7 mm. (height).After finishing, all specimens in each group were glazed using the same previous cooling regimens. The dimensional error in each specimen was accepted at ± 0.01 mm. measured by using a digital veneer caliper.

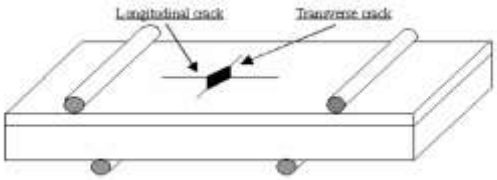


**Figure 1** Schematic illustration of an indented beam specimen placed in four-point flexure. Specimens was indented on the tension surfaced to produced controlled cracks.

**Testing and statistic analysis**

Indentation cracks were induced within the veneering surface of each specimen using Vicker diamond indenter of the microhardness testing machine (FM800, Future-tech, Japan) at a load of 4.9 N for 15 seconds dwelling time to produce controlled cracks. Indentation induced longitudinal and transverse cracks which were measured optically using a reticulated eyepiece at ×50 magnification 24 hours after indentation (Figure1)to allow complete growth of the crack caused by environment degradation as well as residual contact stress. The samples were kept dry at room temperature.

All of the specimens were subjected to the four point bending test with 15 mm. outer span and 5 mm. inner span (crosshead speed of 0.5 mm/min) in a universal testing machine (Lloyd, LR30/k, Leicester, England).The veneer surface was placed in the tension side for all flexure test specimens as shown in figure 3. The load at failure was recorded and flexural stress ($\sigma_f$)for each specimen was calculated using the mechanic and composite beam theory (Taskonak et al., 2005; Beer et al., 2009) using a transformation technique as follows:

1. Determine transformation factor (n)

$$n = E_c/E_v \quad (1)$$

where : -$E_c$ is elastic modulus of zirconia core = 210 GPa

$E_v$ is elastic modulus of IPS e.max CAD = 95 GPa

2. Transformation of bi-layer beam of core and veneer to uniform beam .

$$C = (1/n)(\text{actual width}) \quad (2)$$

3. Determination of centroid tranformed

$$\ddot{y} = \frac{KLM+NOP}{LM+OP} \quad (3)$$

4. Determination flexural stress ($\sigma_f$) in transformed beam of four-point bending

$$\sigma_f = Mc/I \quad (4)$$

where :

$$M = \frac{P_f(L_0-L_i)}{4} \quad (5)$$

$$I = \left\{\frac{1}{12}PO^3 + PO\left(L + \left(\frac{O}{2}\right) - \ddot{y}\right)^2\right\} + \left\{\frac{1}{12}ML^3 + ML\left(\ddot{y} - \left(\frac{L}{2}\right)\right)^2\right\} \quad (6)$$

c =the perpendicular distance to the centroid from the bottom of the transformed beam

In which :

M is the maximum moment, I is the moment of inertia, $P_f$ is failure load, $L_0$ is the outer span = 15 mm., $L_i$ is the inner span = 5 mm.

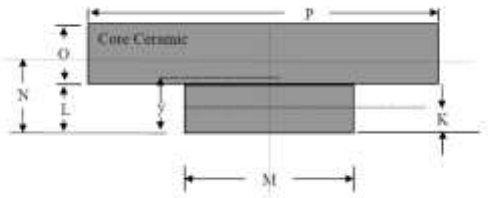


**Figure 2** Transformed and T-shape cross section of specimen

One-Way ANOVA statistical analysis was used (SPSS 19.0, SPSS Inc., Chicago, USA) to verify the significant difference that existed from the effect of the different cooling rate at P-value 0.05. In addition, the reliability of flexural strength was also determined with weibull analysis using the equation as follow:

$$P_f = 1 - exp\left[-\left(\frac{\sigma}{\sigma_0}\right)^m\right] \quad (7)$$

where : -P is the probability of failure, $\sigma$ is the fracture stress at failure, $\sigma_0$ is the characteristic parameter corresponding to the fracture probability of 63.2%, m is the weibull modulus

**Results**

Table 2 and figure 4 represent the results of the four point bending test in terms of the mean and standard deviation (X±SD).The means of the flexural strength of fast, medium and slow cooling were 920.93±89.71, 912.88±62.51, 892.42±60.75 MPa respectively. An analysis of variance (One- Way ANOVA) indicated that there was no statistical significant difference (P>0.05) on the means flexural

**Table 1** Firing parameters for CAD-on technique

| Program | B ($^0$C) | S (min) | $t_1$ ($^0$C/min) | $T_1$ ($^0$C) | $H_1$ (min) | $t_2$ ($^0$C/min) | $T_2$ ($^0$C) | $H_2$ (min) | L (Fast Cooling) ($^0$C) | L (Medium Cooling) ($^0$C) | L (Slow Cooling) ($^0$C) | $V1_1$/$V1_2$ ($^0$C) | $V2_1$/$V2_2$ ($^0$C) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Crystallization | 403 | 02.00 | 30 | 820 | 02.00 | 30 | 840 | 07:00 | 600 | 560($T_g$) | 200 | 550/820 | 820/840 |
| Glazing | 403 | 6.00 | 60 | 820 | 0:10 | 30 | 840 | 03:00 | 600 | 560($T_g$) | 200 | 550/820 | 820/840 |

*NB: B= Stand by temperature; S=Closing time; $t_1$, $t_2$= heating rate; $T_1$,$T_2$=Firing temperature;$H_1$, $H_2$=Holding time; L= Long term cooling*

**Table 2** Mean, standard deviation of flexural strength and weibull modulus

| Group | Flexural strength | | | | | | Weibull modulus |
|---|---|---|---|---|---|---|---|
| | Mean (MPa) | SD | Max (MPa) | Min (MPa) | 95% CI Lower bound | 95% CI Upper bound | |
| Fast | 920.93 | 89.71 | 1074.90 | 816.52 | 869.14 | 972.73 | 11.75 |
| Medium | 912.88 | 62.51 | 1069.98 | 788.46 | 876.79 | 948.97 | 16.88 |
| Slow | 892.42 | 60.75 | 1023.44 | 800.33 | 857.34 | 927.49 | 17.24 |

**Table 3** One-Way ANOVA test results

| Stress | SS | Df | MS | F | P |
|---|---|---|---|---|---|
| Between groups | 6049.744 | 2 | 3024.872 | .580 | .565 |
| Within groups | 203386.473 | 39 | 5215.038 | | |
| Total | 209436.217 | 41 | | | |

strength among the tested groups due to the different cooling rates as shown in Table 3. The flexural strength in the fast cooling group has the highest value (920.93 MPa) whereas the slow cooling group has the lowest value (892.42 MPa). The weibull analysis was shown in table 2 and figure 5, the slow cooling group represents the highest weibull modulus (17.24) and the fast cooling group shows the lowest (11.75).

**Discussion**

The present study assessed whether the cooling rate had an influence on the flexural strength of the CAD-on veneering zirconia. The results accept the null hypothesis. In this study, the cooling rate was set by using glass transition temperature as a variable when setting the firing programs, the temperature for the muffle opening is higher, at, or lower than the glass transition temperature of veneering glass. All of the specimens were fired according to the cooling regimens in both sintering and glazing processes, and left in the muffle until the ambient temperature was reached. The findings of this study are different to those of prior studies. Tan et al. (2012) suggested to use slow cooling regimen when firing porcelain fused to zirconia prostheses. Nevertheless, in their study, the muffle was open when the temperature was higher than the glass transition temperature in all of the specimens firing cycles. Komine et al. (2010) concluded that the duration of cooling from firing temperature to room temperature may affect the shear bond strength of veneering porcelain to a zirconia

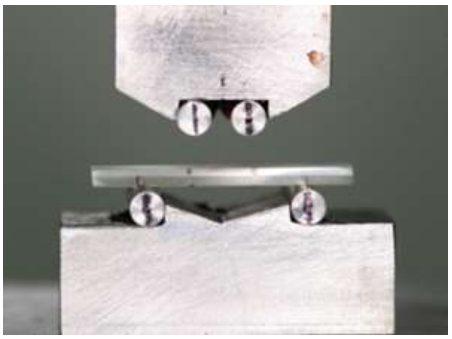

**Figure 3** Four point bending test apparatus with a specimen in place

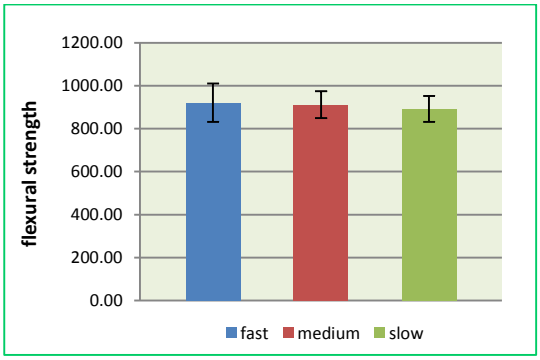


**Figure 4** Comparing mean flexural strength of fast, medium and slow cooling groups

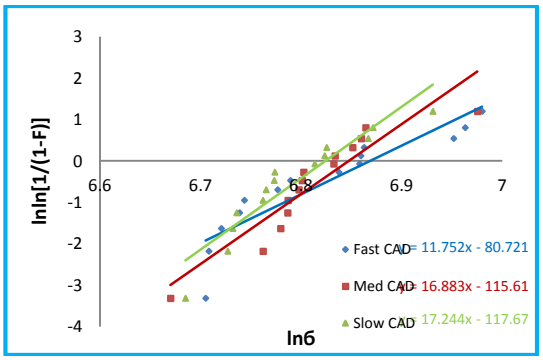


**Figure 5** Weibull analysis of fast, medium and slow cooling groups

material depending on the porcelain material used. Although their study supported using the slow cooling regimen, all of the specimens in their study were cooled in the muffle from firing temperature to the glass transition temperature before opening the muffle. Moreover, there were only two cooling regimens : removing the specimens immediately from the furnace and cooling in ambient air or letting the specimens cool down beside the muffle for four minutes, and the shear bond strength did not have statistical significance different due to the difference of cooling time in some veneering glass material. On the other hand, Göstemeyer et al. (2010), Taskonak et al. (2008) suggested to use fast cooling in order to generate superficial compressive residual stresses in zirconia/glass ceramic fabrications. They explained that the compressive residual stresses may compensate to the tensile stresses that were generated at the surface by loading tests. However, the cooling regimens reported by Taskonak et al. were only applied in the heat treatment program. In addition, all of the studies that were mentioned above used the conventional layering technique that had to use multiple firing programs due to the condensation processes. Unfortunately, Zeighami el al. (2013) stated that the coefficient of thermal expansion mismatch between zirconia core and veneering ceramic controlled by the manufacturers can produce residual stress in the core/veneer interface when the multiple firing occur and it could be the cause of the reduction in fracture strength of the veneering ceramic. Therefore, an importance of cooling rate in the conventional layering technique is the way to optimize the residual stress especially when the multiple firing cannot be avoided. Nonetheless, there has been no report about the influence of cooling rate when using CAD-on veneering ceramic corresponding with zirconia substructure. From the results of this current study that the flexural strength of CAD-on veneering ceramic did not affect by the different cooling rates. A possible explanation might be the decreasing of the firing cycles when using the pre-crystallized veneering ceramic that is consistent with the study of Zeighami el al.. Besides the outstanding in mechanical properties of pre-crystallized lithium disilicate glass ceramic introduced into CAD-on veneering technique, an interesting advantage of this technique is the reducing firing process as it needs only one firing cycle in order to fuse the milled veneering cap to the complete sintered zirconia substructure. When the process needs less firing cycle, it could be assumed that the less residual stress

occur, and it could be implied that the cooling rate which is the factor that generate the residual stress may has less influence on fracture strength of the restorations. In other words, the strength of the restoration fabricated from this technique may not rely on the cooling rate.

**Conclusion**

Within the limitation of the present study,it could be concluded that the different cooling rate may not affect the flexural strength when using the CAD-on veneering technique, and further studies are required to clarify the effects of the cooling rates on this veneering technique.

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