Evaluation of the shear bond and bi-axial flexural strength of zirconia core veneered by CAD/CAM and PRESS-ON technique

*Mohamed Al basher Zeglam **Milad Mustafa Eshah
***Seham Ali Elsawaay ****Mohamed Abdalla Belead

Abstract
The aim of this in vitro study is to compare the shear bond and bi-axial flexural strengths of samples veneered with, CAD/CAM-fabricated zirconia core, and Press-on technique, with the definition its mode of failure.

Twenty zirconia core discs were made using IPs e.max ZirCAD and CAD/CAM technology. These were divided equally in to two groups. Group one was veneered by CAD-on technique using IPs e.max CAD, and group two was veneered by Press-on technique using IPs e.max ZirPress, then all the discs are thermocycled. The specimens were exposed to shear bond and biaxial flexural strength testing. Mode of failure was evaluated by SEM. All data were collected and analyzed statistically by SPSS, with significance level 5%, where \( P \leq 0.05 \). The t- test analysis of shear strength test showed statistically significant difference at \( (p < 0.05) \) for CAD-on group. While there were no statistically significant difference at \( (p < 0.05) \) for the biaxial flexural strength test between the two groups. CAD-on group showed adhesive/cohesive failure, while press-on group showed cohesive failure within the veneering ceramic. This study concluded that, the bond strength between zirconia core and veneer was affected by the veneering technique. CAD-on technique showed statistically significant high bond strength over Press-on technique

Key words: Zirconia, Shear Bound, Flexural Strength, CAD/CAM, PRESS ON

* MDs in Fixed prosthodontics -Tripoli University- Faculty of Dentistry- Libya
** DDs in Fixed prosthodontics -Tripoli University- Faculty of Dentistry- Libya
*** MDs in Fixed prosthodontics -Tripoli University- Faculty of Dentistry- Libya
**** MDs in Fixed prosthodontics -Misruta University- Faculty of Dentistry- Libya
INTRODUCTION

The demands of all ceramic dental prosthesis have increased widely in the last 19th century, and the advanced processing technology of these materials makes it of superior interest for patient, dentist and technician (Kelly et al, 1995). Aesthetic, strength and biocompatibility of Zirconia base material improved its used for both crowns and fixed partial dentures. Among different types of ZrO2 ceramics, the most frequently used material is Yttria-stabilized Tetragonal Zirconia Polycrystal (Y-TZP), which had been introduced in the early 1990s. Compared to other core ceramics, Y-TZP ceramic demonstrates better mechanical properties and a superior resistance to fracture. Yttrium-oxide (Y2O3 3% mole) is added to pure zirconia to control the volume expansion and to stabilize it in the tetragonal phase at room temperature (Denry et al, 2008). Zirconium dioxide-based ceramics (ZrO2) has been applied to clinical use through the computer-aided design/computer-aided manufacturing technologies (CAD/CAM) (Larsson et al, 2014). Either the layering technique or the press-over technique can be used for veneering the zirconia cores. The layering technique allows superior aesthetic results compared with the press-over technique, which has the advantages of being faster and less costly (Eisenburger et al, 2011). Beuer, 2009 introduced a new procedure for veneered all-ceramic crown restorations using a CAD/CAM-fabricated high-strength zirconia coping and a corresponding CAD/CAM-fabricated lithium-desilicated glass-ceramic veneering material. It can be assumed that this procedure of sintering core and veneering leads to an increase in mechanical strength compared to traditional techniques enabling a lower clinical chipping rate of the veneering material due to the use of CAD/CAM generated high-strength veneering ceramics. The aim of this in vitro study was to Evaluate the shear bond and bi-axial flexural strengths of CAD/CAM-fabricated zirconia core veneered with CAD/CAM-fabricated lithium-desilicated glass-ceramic veneering material (CAD-on) as compared with press-on technique, and to Evaluate its mode of failure

MATERIAL AND METHODS

This study included two groups, one group used CAD-on veneering technique, and the other group used Press-on veneering technique for zirconia core. Each group was subjected to two types of testing, which were shear
bond and biaxial flexural strength tests. The specimens for shear bond strength test were in a form of zirconia core discs (5 mm diameter ×4 mm height), veneered with veneering ceramic discs (3 mm diameter × 3 mm height), 5 each group. The specimens for biaxial flexural strength test were in a form of zirconia core discs (12 mm diameter × 0.7 mm height), veneered with veneering ceramic discs (12 mm diameter × 0.8 mm height), 5 each group.

Specimens’ preparation for shear bond strength test:
(A) Core fabrication: ten Zirconia core discs were constructed (5×4) mm from IPS e.max ZirCAD blocks. And were divided in to two groups, five each group, group one were veneered by CAD on technique using , group two were veneered by Press on technique.
(B) Veneer fabrication:
CAD-on technique: five veneer discs (3×3) mm were constructed from Ips e.max CAD blocks. The sintered zirconia discs were fused to the milled Ips e.max CAD discs by low fusing ceramic (Ips e.max cad crystal/connect fusion glass ceramic), which came in a form of capsule that was put under vigorous vibrations through Ivomix device that converted it in to applicable form, the discs were then entered fusing/crystallization firing cycle in ceramic furnace (programat p 300), using the following parameters;

Table (1): fusion/crystallization parameters (°c)

<table>
<thead>
<tr>
<th>B</th>
<th>S</th>
<th>t1</th>
<th>T1</th>
<th>H1</th>
<th>t2</th>
<th>T2</th>
<th>H2</th>
<th>v₁₁ / v₁₂</th>
<th>v₂₁ / v₂₂</th>
<th>L</th>
</tr>
</thead>
<tbody>
<tr>
<td>403</td>
<td>2:00</td>
<td>30</td>
<td>820</td>
<td>2:00</td>
<td>30</td>
<td>840</td>
<td>7:00</td>
<td>550/820</td>
<td>820/840</td>
<td>600</td>
</tr>
</tbody>
</table>

Where:

B: starting temperature [°c]
S: closing time [min]
t₁ : heating rate, phase 1 [°c/min]
T₁: firing temperature, phase 1 [°c]
H₁: holding time, phase 1 [min]
t₂ : heating rate, phase 2 [°c/min]
T₂: firing temperature, phase 2 [°c]
H2: holding time, phase 2 [min]
V1: vacuum on
V2: vacuum off
L: long-term cooling [°C]

Press-on technique: zirconia discs were prepared via sandblasted with 50 μm Aluminum oxide (Al₂O₃) and application of IPS e.max Ceram ZirLiner to achieve a sound bond between zirconia discs and IPS e.max ZirPress veneering material. Waxing up and investing done by using double diameter copper mold sizes ((4x5), (3x3)) mm, zirconia discs were put on the larger size diameter space, a sprue wax (3x3) mm was fixed on it and completed spruing on investment ring (100g), and invested according to manufacturer’s instructions, then entered the burn out furnace for wax burn out according to manufacturer’s instructors. Pressing is done using a cold IPS Alox Plunger and IPS e.max ZirPress ingots in the desired shade, which were placed into the hot investing ring, with shade imprint facing upward, the investing ring was then put in the center of the hot press furnace (programat EP 3000), and pressing was done according to the parameters described in table (2). After the press program has been completed, the ring was allowed to cool down to room temperature.

**Table (2): Press program parameter (°C)**

<p>| | | | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>t</td>
<td>T</td>
<td>H</td>
<td>E</td>
<td></td>
</tr>
<tr>
<td>700</td>
<td>60</td>
<td>900</td>
<td>15:00</td>
<td>300</td>
<td></td>
</tr>
</tbody>
</table>

B: Starting temperature [°C]

\[ t \]: Heating rate [°C /min]

T: Firing temperature [°C]

H: Holding time [min]

E: end of program (um/min)

After cooling the length of the Alox plunger was marked on the cooled investment ring. This predetermined breaking points enabled reliable separation of the investment material and the discs. A separating disk was used to separate the investment ring. Polishing beads were used to clear the
pressed discs. The reaction layer on the surface of the discs was removed by immersing them on IPS e.max Press Invex Liquid for 30 minute according to manufacturer’s instruction. Then were rinsed with running water and dried, and final finishing was done. Glaze was applied and fired according to manufacturer’s instructions.

Specimens preparation for biaxial flexural strength test:

Core fabrication: ten Zirconia core discs were constructed (12×0.7) (Guess et al, 2013) mm from IPS e.max ZirCAD blocks. And were divided in to two groups, five each group, group one were veneered by CAD on technique using, group two were veneered by Press on technique.

Veneer fabrication: CAD-on technique: using the same mention technique before but the size of the veneer disc is (12×0.8) mm.

Press-on technique: using the same mention technique before but the size of the veneer disc is (12×0.8) mm. The test specimens were subjected to thermocycling machine

for 600 cycles (corresponding to one year), at 55° C and 5° C with a dwell time (time taken by the specimens in each bath) of 2 minute, and transfer time of 10 seconds.( Krejci et al, 1994) . after thermocycling the specimens were subjected to shear bond strength and biaxial flexural strength as followed Shear bond strength test: each specimen fixed in acrylic mold and secured to the lower plate of universal testing machine *by specially designed jig. The knife edge blade of the universal testing machine was held against the zirconia /ceramic veneer interface and a shear force was applied to each specimen at a cross-head speed of 1 mm/min until failure occurred. The load at which the failure occurred was recorded in Newton ( Özkurt et al ,2010). The Shear bond strength was calculated according to the following equation( Özkurt et al ,2010)

Shear stress (MPa) = Load (N) / (Πr²) N/mm²

Where, Π = 3.14    r = radius of the specimen    N = load in Newton

Selected fractured specimens were examined under SEM , to evaluate the mode of failure. Biaxial flexural strength test: each disc was fixed to the lower plate of universal testing machine by a specially designed jig, in which each disc was placed on a circular 10 mm diameter knife-edge support and the load was applied centrally at a rate of 1mm\minute with a spherical indentor of 5 mm (Wakabayashi et al ,2000) . During testing, all the specimens were
placed with the glazed veneer surface facing upward toward the indentor and a thin sheet of rubber was placed between the disc and the knife-edge support to accommodate slight irregularities in the disc (Wakabayashi et al, 2000).

The biaxial flexural strength was calculated according to the following equation (Guess et al, 2009).

\[
T = \frac{WV(a-b)}{Rd}
\]

\[
R = 0.675d
\]

Where:
- \(T\) is the maximum tensile stress
- \(W\) is the measured load at fracture
- \(V\) is the Poisson’s ratio for the material
  - (A value of 0.25 was substituted for the material)
- \(R\) is the equivalent radius of loading
- \(A\) is the radius of the knife-edge support
- \(B\) is the radius of the tip of the piston
- \(d\) is the thickness of specimens

**Statistical Analysis:** All data were collected and analyzed statistically by using SPSS program software (Statistical Package for Social Science- version 15.0), with significance level 5%, where \(P \leq 0.05\).

**Results**

**Results of Shear bond strength test:**

<table>
<thead>
<tr>
<th></th>
<th>CAD-on</th>
<th>Press-on</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>10.65 MPa</td>
<td>5.89 MPa</td>
</tr>
<tr>
<td>Max</td>
<td>18.48 MPa</td>
<td>7.01 MPa</td>
</tr>
<tr>
<td>Mean ± SD</td>
<td>13.49± 3.622</td>
<td>6.44± 0.411</td>
</tr>
<tr>
<td>t value</td>
<td>4.325</td>
<td></td>
</tr>
<tr>
<td>P value</td>
<td>0.003</td>
<td></td>
</tr>
</tbody>
</table>

*\(t\) and \(P\) values were calculated using Student \(t\)-test*
Evaluation of the shear bond and bi-axial flexural strength of zircona core veneered by CAD/CAM and PRESS-ON technique

Results of Biaxial flexural strength test:

<table>
<thead>
<tr>
<th></th>
<th>CAD-on</th>
<th>Press-on</th>
</tr>
</thead>
<tbody>
<tr>
<td>Min</td>
<td>52.60MPa</td>
<td>26.96MPa</td>
</tr>
<tr>
<td>Max</td>
<td>150.14MPa</td>
<td>121.29MPa</td>
</tr>
<tr>
<td>Mean ± SD</td>
<td>84.93 ± 38.51</td>
<td>55.57 ± 37.45</td>
</tr>
<tr>
<td>t value</td>
<td>1.22</td>
<td></td>
</tr>
<tr>
<td>P value</td>
<td>0.26</td>
<td></td>
</tr>
</tbody>
</table>

\( t \) and \( P \) values were calculated using Student t-test

Figure (1): Mean value of shear test of the two studied groups.

Figure (2): Mean value of biaxial flexural strength of the two test groups.
Result of Mode of failure, CAD-on group: all of the five specimens showed adhesive/cohesive failure, which contributed to 100% of the total specimens in this group. Press-on group: all of the five specimens showed cohesive failure within the veneering ceramic, which contributed to 100% of the total specimens in this group.

Discussion

(Ereifej et al., 2011) stated that the bond strength and the quality between the veneer and the ceramic core are the key factors in the success of bilayered restorations. In the current study, yttrium oxide partially stabilized zirconia (Y-TZP) was selected as a core material. It is industrially manufactured into blanks and milled to the desired dimensions using (CAD/CAM) technology. Cores made in this way are stable, have a high crystalline content and flexural strength of 900–1200Mp (Tsalouchou et al., 2008). The combination of a CAD/CAM-fabricated framework with CAD/CAM-fabricated veneering would be of major interest, especially if considerably stronger veneering ceramics can be applied (Beuer et al., 2009). Lithium-disilicate reinforced glass-ceramic (IPS e.max CAD) is one of the veneering ceramics that are used for zirconia framework. And since it is industrially produced in to ceramic blanks, it shows fewer faults compared to manually produced veneers (Beuer et al., 2009). Thus, the fatigue behavior and the fracture strength are supposed to be superior in these materials compared to hand-layered ceramics (Guess et al., 2009). Additionally, the material properties of IPS e.max CAD ceramics (flexural strength: 360 MPa) are superior compared to those of hand-layered feldspathic ceramics (flexural strength about 100 MPa) (Schmitter et al., 2012). This ceramic is suitable for the sinter bond technique with zirconia frameworks due to the appropriate match of the coefficients of thermal expansion (CTE of IPS e.max ZirCAD= 10.75×10−6 K−1, CTE of IPS e.max CAD= 10.25×10−6 K−1) (Beuer et al., 2009). These reasons might explain the use of this material in the present study. It was reported that zirconia veneer bond strength is affected by veneering techniques (Blatz et al., 2010). An advance veneering category is CAD-on technique, which was used in this study, it includes that both the framework and the veneer are CAD/CAM manufactured, resulting in almost faultless components as the ceramic blanks are industrially produced (Schmitter et al., 2012). After the manufacturing of the framework and the veneer, the components are joined
together using fusion glass ceramics in a special programmed furnace (Kanat et al., 2014). This technique combined both strength and esthetic (translucency) of restoration. However, little information is available about the behavior of these completely CAD/CAM-manufactured restorations (Kanat et al., 2014). Heat pressing materials have become available to produce a veneering layer for all ceramic restorations (Choi et al., 2011). And has shown improved mechanical, fatigue properties and clinical performance of all ceramic restoration (Tsalochou et al., 2008). IPS e.max ZirPress is a fluorapatite glass-ceramic ingot, it has an appropriate coefficient of thermal expansion with respect to zirconia. (IPS e.max ZirPress= 9.75±0.25×10−6 K−1), (IPS e.max ZirCAD (10.75×6−1)· K−1) (Beuer et al., 2009). With a flexural strength of (110 ± 10 MPa). These reasons might explain the use of this material in this study. Press-on technique is a veneering category used in this study, it combines both performance improvement and better bonding to zirconia framework as compared with conventional layering (Blatz et al., 2010). Press-on technique depends on lost wax technique, where the required shape and form of the veneer ceramic is obtained under a control temperature, pressure and vacuum parameters, which reduces the possibility of thermal fatigue and incorporation of structural defects, although wax design still remains operator dependent and a factor of human error exist (Kelly et al., 1995). In the current study, standardization of the specimens of each test group was done by producing them from the same standard molds, with the same dimensions. In this study, a thermocycling regime was conducted to simulate intra-oral temperature changes. According to (Krejci et al., 1993), 600 cycles corresponded to one year of clinical performance. In the present study, shear bond strength test (SBS) was used to determine the core veneer bond strength. In the (SBS) test the stresses determined for each specimen are the best normal approximation of the actual stresses applied on fixed partial dentures at the location of failure (Kelly et al., 1995). Also, the failure process under a knife edge is similar to the stress concentration effect on the occlusal contact (Smith et al., 1994). On the other hand it resulted in more standardized data, because the applied forces are perpendicular to the bonding area, and the small cross-sectional area of the bonded surface eliminates incorporation of structural flaws, which significantly affect test readings (Blatz et al., 2010). In this study biaxial flexural strength test (BFS) was also used, it was added to the ISO 55
standard for dental ceramics, the test provides an equal biaxial stress
distribution, and a maximum tensile stress (Ceramic materials ISO , 2008)
occurs at the central loading area rather than at the edges (Kelly et al, 1999). It
has been introduced to measure the tensile strength of brittle materials such as
ceramics, because the disc shape specimens are more close in size and volume
for dental restorations than bares used for other tests (Ceramic materials ISO,
2008). Also the biaxial strength test is well known to induce a very complex
state in the test specimens and thereby simulating the intra-oral situation
encountered by the restoration (Cattell et al, 1997). A wide variety of loading
arrangements have been developed for biaxial flexure tests, one of them is
ball-on-ring test which was selected in this study (Borba et al, 2011) .
Regarding shear bond strength, the results revealed that, there was a significant
difference in the shear bond strength between the CAD-on and Press-on
techniques, where CAD-on technique demonstrated significantly higher shear
bond strength than press-on technique. This might be attributed to the methods
of fabrication of these two ceramic systems. In CAD-on group a digitalization
was used in fabrication of the specimens which might had influence on its
bond strength. Digitalization result in accurate sample size and dimension,
which affect precise fit and adaption between the core and veneer. And a very
thin and uniform layer of fusing ceramic is thus obtained. This fusing ceramic
had high fusion capability and flow properties which are enhanced by vigorous
vibration (230 Hz), in addition the excellent wetting ability of zirconia would
enable some degree of penetration of the fusing ceramic along the Y-TZP
boundaries. Vigorous vibration also help to eliminate structural defects such
as air bubbles, voids and micro-gaps, thereby eliminating stress concentration
sites. The fusing ceramic had a matched Coefficient of thermal expansion to
both IPS e.max- ZirCAD core and the IPS e.max CAD veneer, and as it
entered with the veneer the same cycle with high temperature, this result in
completely homogenous fusion and bonding from both sites resulting in
compressive bond between the core and the veneer. Also the fusing ceramic
and IPS e.max CAD veneer were allowed to cool at the same cooling rate and
this might decreased the cooling stress. All these reasons might explain the
high bond strength obtained in CAD-on group, Press-on techniques, showed
lower bond strength than CAD-on technique, demonstrating that adhesion of
IPSe.max ZirPress to zirconia was low. This might be attributed to the
difference in the structure and fabrication techniques used for both core and veneer, as zirconia core was CAD/CAM fabricated, while the veneer was heat pressed. This result is consistent with (Choi et al., 2011) who found that non-leucite containing glass ceramic had less adhesion to zirconia than leucite contain glass ceramic, and IPSe.max ZirPress is a non leucite contain glass ceramic. (Gelman et al., 2009) also found that pressed veneering ceramic revealed bond strength values to zirconia that were not different from layered ceramics. Regarding mode of failure in the current study, the results revealed that, the specimens displayed adhesive/cohesive failure in CAD-on group, while it showed cohesive failure limited within the veneer material in Press-on group. This result is in accordance with (Özkurt et al., 2010), who observed adhesive/cohesive failure in the zirconia base ceramics they used. Press-on group showed cohesive failure, and this might be due to the nature of pressed veneer ceramic, since it was weaker and easily delaminated compared to the high strength CAD-on veneer. This result is in agreement with (Tsalouschou et al., 2008) who found mainly cohesive failure within the pressing veneer materials they used. Regarding biaxial flexural strength, the results revealed that, no statistically significant difference between CAD-on and Press-on techniques. This result might be related to microstructural nature of the supported zirconia core material. This result is consistent with (Lin et al., 2012) who found that different fabrication techniques (heat-pressed and CAD/CAM) have no effect on the biaxial flexural strength. (Yilmaz et al., 2011) showed a similar biaxial flexural strength recorded at the bottom surface of bilayer zirconia specimens. (Eisenbuger et al., 2011) found no significant different in fracture load between layering and press-on techniques when zirconia core was used. Similarly, anthers in vitro studies done by (Guess et al., 2009) and (Stawarczyk et al., 2011) revealed the same result. Ceramic structures tend to fail because of surface tension, where cracks and flaws propagate by slow crack growth leading to the catastrophic failure (Beure et al., 2009). Catastrophic failure as a result of contact loading has made it difficult to identify whether cone cracking or subsurface damage was responsible, it is supposed that both processes may occur at the failure site (Beure et al., 2009). In all ceramic systems, the flaw population (size, number and distribution) can be related to the material, or affected by the fabrication process. Thus, it might be expected that heat pressing and sintering might
introduce significantly less flaws, resulting in a good strength properties, as both techniques are controllable procedures (lin et al ,2012) . Industrially produced ceramic blanks used for the fabrication of veneers show fewer faults compared to manually produced veneers (Schmitter et al, 2012). CAD/CAM process uses a high quality material with a minimum of flaws. Although there is a lack of studies assessing the fracture load of zirconia restorations veneered using CAD/CAM machined lithium disilicate ceramics, there are studies confirming that lithium disilicate ceramic CAD/CAM crowns may be an effective option for all-ceramic crowns (Siervo et al , 1994). (Schmitter et al 2012) demonstrate that crowns made of zirconia frameworks veneered with CAD/CAM manufactured lithium disilicate ceramics display a higher fracture resistance than manually veneered crowns. (Choi et al, 2011) stated that no systematic investigations of flexural strength of pressed ceramic to zirconia are available. Compared with the conventional powder/liquid layering technique for zirconia core material, (Aboushelib et al, 2008) showed that heat-pressed veneering porcelain produces significantly higher fracture strength when pressed to zirconia core. Also (Lin et al, 2012) revealed that heat-pressed technique provided significantly high flexural strength for the zirconia bilayer specimens. (Fischer et al ,2008) stated that, to realize the benefit of high strength zirconia as a framework, veneering ceramic has to strengthen.

**Conclusion:**

Bond strength between zirconia core and veneer was affected by the veneering technique. CAD-on technique showed statistically significant high bond strength over Press-on technique

**Reference**

4. Beuer F, Schweiger J, Eisenburger M, Kappert HF, Gernet W, Edelhoff D. High-strength CAD/CAM-fabricated veneering material sintered to zirconia copings -
17. Kelly JR, Nishimura I, Campbell SD. Ceramics in dentistry: historical roots and

60