A focus on zirconia: an in-vitro lifetime prediction of zirconia dental restorations
Rosentritt, M.E.

Citation for published version (APA):

General rights
It is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), other than for strictly personal, individual use, unless the work is under an open content license (like Creative Commons).

Disclaimer/Complaints regulations
If you believe that digital publication of certain material infringes any of your rights or (privacy) interests, please let the Library know, stating your reasons. In case of a legitimate complaint, the Library will make the material inaccessible and/or remove it from the website. Please Ask the Library: http://uba.uva.nl/en/contact, or a letter to: Library of the University of Amsterdam, Secretariat, Singel 425, 1012 WP Amsterdam, The Netherlands. You will be contacted as soon as possible.
Influence of ceramic veneering on the fracture of zirconia FPDs.

Keywords: zirconia, CAD/CAM, veneering, fracture resistance, artificial aging
4.1 Abstract

Objective. Clinical data show good experience with zirconia FPDs, but clinical failures due to chipping of the ceramic veneering occur. The aim of this study was to compare the fracture resistance of zirconia cores with various veneering after thermal cycling and mechanical loading (TCML).

Materials and Methods. Sixty-four three-unit zirconia FPD frameworks (Cercon Base, Degudent) were milled and veneered with different layering ceramic variations. Thermal expansion coefficient (TEC; 8.7 - 9.9 μm/mK) and firing temperature (FT; 750 – 830 °C) were varied. As a reference, porcelain fused to metal three-unit FPDs was used. All restorations were adhesively luted to human molars, thermally-cycled with synchronous mechanical loading, and finally fractured in a universal testing machine. To investigate the influence of the artificial aging, one zirconia group was loaded without aging.

Results. TCML reduced the median fracture force from 1735N to 1227N. All-ceramic zirconia FPDs with ceramic veneering showed no significant differences compared with the porcelain fused to metal reference. The fracture force increased with increasing thermal expansion coefficient and firing temperature from 8.7 μm/mK / 750 °C (533 N), over 9.3 μm/mK / 780 °C (1098 N) to 9.7 μm/mK /830 °C (1227 N). A significant Pearson correlation was found between fracture force and firing temperature (p = 0.039) but not between force and TEC (p = 0.067).

Conclusion. To avoid chipping, TEC and FT of the veneering need to be adapted to the zirconia framework.
4.2 Introduction

High-strength zirconia core materials are well known in medical applications (e.g. hip joints), but only limited evidence-based data is available for their dental use [1-4]. In comparison with glass ceramics, polycrystalline yttria tetragonal zirconia (Y-TZP) shows three to four times higher flexural strength (800-1200 MPa) and flexural toughness (>10 MPa x m\(^{1/2}\)). Therefore, it is preferred as the core material for all ceramic restorations in stress-bearing posterior areas. Most zirconia ceramics need to be processed by computer-aided manufacturing (CAM), in combination with computer-aided design (CAD). They are milled in the hot isostatic pressed or partially-stabilized pre-sintered state. Milled zirconia has high fracture strength with a small range of strength variation and high structural reliability compared with conventional dental ceramics [5, 6].

Zirconia cores promise high-strength restorations, but veneering with “weaker” conventional glass-ceramics (with crystalline phases), which are used for aesthetic appearance, function and protection, may influence the resistance to fracture of the restoration in service [7]. Zirconia cores may be veneered with various firing ceramics (Sakura, Ceramco, Cercon Kiss, Cercon Ceram S) or pressable alternatives (Cercon Ceram Express), but an inappropriate combination of core and veneer may show unpredictable failure of the veneer [8]. This may be a reason for the clinically reported chipping of up to 15% in zirconia FPDs [1-4]. In-vitro bond strength investigations between core and veneering have shown the influence of the thermal expansion coefficient, zirconia transformation, and sintering temperature on the integrity of the zirconia veneer interface [9-11]. The influence of veneer thickness [12] and core pre-treatment [13] has been discussed. The failure mode between zirconia and veneer is supposed to be predominantly interfacial [9, 14], but exceptions were found on fixed partial dentures (FPDs) [8]. During mechanical loading, local occlusal stress may superpose with global compressive residual stress resulting in lateral cracks and chipping [15]. The veneering ceramic with a small reliability (Weibullmodul m ~ 1 MPa x m\(^{1/2}\)) may be associated especially with pre-existing bulk or surface defects [16].

Significant decisions for evaluating the usability of dental materials or restorations have been made using evidence-based information, but clinical studies are cost- and time- expensive. Artificial oral environments, which combine thermal cycling with mechanical loading (TCML), are used for the prompt and cost-effective estimation of the usability of dental reconstructions. At least, they may help to avoid catastrophic failure of the restoration in service [17].

The aim of this study was to evaluate the influence of various veneer material properties on the fracture resistance of three-unit zirconia FPDs. An artificial mouth was used for a time-lapsed aging of the restorations, simulating the influence of chewing force and temperature on
the FPDs. Restorations were monitored during aging, and fracture resistance was determined after aging. The results after TCML were compared with available clinical data.

4.3 Materials and Methods

The roots of human molars were coated with a 1 mm thick polyether layer (Impregum, 3M-Espe, Seefeld, Germany) to simulate human periodontium. Loaded to 50 N, the layer allows a maximum mobility of the single tooth in axial and vertical directions of 0.1 mm. Two teeth were inserted into polyethylene-methacrylate resin (Palapress Vario, Kulzer, Wehrheim, Germany) forming a gap of 10 mm. Human molars were used to ensure clinical conditions including the mechanical performance of the abutments and bonding between FPD and tooth. Varying dimensions of the teeth were therefore accepted.

All teeth were prepared according to the directives for ceramic restoration techniques, using a 1 mm deep circular chamfer preparation. Sixty-four zirconia ceramic three-unit FPDs cores (Cercon Base, DeguDent GmbH, Hanau, Germany) were fabricated according to the manufacturer’s instruction. The cross-section of the connector was 12 mm² (height: 4 mm). The yttria-stabilized zirconia Cercon cores were milled in “white” ceramic condition (Cercon Brain milling machine) and sintered to final dimensions (Cercon Heat oven). This procedure is a time- and tool-saving fabrication process in comparison with milling of sintered high-strength zirconia ceramics. Frameworks were veneered with various ceramic materials using a conventional layering technique. The veneering ceramics varied in thermal expansion coefficient (8.7 - 9.7 μm/mK) and firing temperature (750 - 830 °C). Two materials from different manufacturers were used (Table 4.1). As a reference (#1), eight FPDs were fabricated in a gold alloy (Degudent H, DeguDent) and veneered with a layering ceramic (Duceram Plus, DeguDent). The thickness of all veneering was in a clinically relevant range of 0.5 to 1.5 mm. Liner was used in all cases. To investigate the influence of TCML, one zirconia group with the recommended veneering was investigated without aging (#0). All FPDs were adhesively luted to the abutment teeth using the dual curing composite cement Variolink II (high viscosity) and the dentin adhesive system Syntac classic (both Ivoclar-Vivadent, Schaan, Liechtenstein) after total etching. The fitting of the FPD abutments was checked (Silasoft, Detax, Ettlingen, Germany), and the FPDs were adjusted and sandblasted with 50 μm / 2 HPa.
Table 4.1: Materials, manufacturer, thermal expansion, firing temperature and results (fracture results; pattern).

<table>
<thead>
<tr>
<th>#</th>
<th>0</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Comments</td>
<td>Reference</td>
<td>without TCML</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Veneering material</td>
<td>Reference</td>
<td>PFM</td>
<td>Cercon Ceram S</td>
<td>Duceram</td>
<td>Cercon Ceram</td>
<td>experimental</td>
<td>experimental</td>
<td>Cercon Ceram S</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Plus</td>
<td></td>
<td></td>
<td></td>
<td>Cercon Ceram</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Kiss</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Sakura Interaction</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Ceramco PFZ</td>
</tr>
<tr>
<td></td>
<td>Core materials</td>
<td>Cercon Base</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Degudent H</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Manufacturer</td>
<td>DeguDent GmbH, Germany</td>
<td></td>
<td></td>
<td>Elephant, NL</td>
<td></td>
<td></td>
<td></td>
<td>Ceramco, USA</td>
</tr>
<tr>
<td></td>
<td>Thermal expansion TEC</td>
<td>9.5</td>
<td>13.0</td>
<td>8.7</td>
<td>9.7</td>
<td>9.3</td>
<td>9.5</td>
<td>9.2</td>
<td>9.9</td>
</tr>
<tr>
<td></td>
<td>[μm/mK] 25-500°C</td>
<td>10.5</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Firing temperature FT</td>
<td>830</td>
<td>910</td>
<td>750</td>
<td>760</td>
<td>780</td>
<td>830</td>
<td>830</td>
<td>920</td>
</tr>
<tr>
<td></td>
<td>[°C]</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>900</td>
</tr>
<tr>
<td></td>
<td>Fracture force median</td>
<td>1735</td>
<td>1329</td>
<td>533</td>
<td>1236</td>
<td>1098</td>
<td>1227</td>
<td>1331</td>
<td>1440</td>
</tr>
<tr>
<td></td>
<td>[N]</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1380</td>
</tr>
<tr>
<td></td>
<td>25%</td>
<td>1305</td>
<td>1218</td>
<td>436</td>
<td>976</td>
<td>857</td>
<td>1115</td>
<td>1224</td>
<td>1294</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>940</td>
</tr>
<tr>
<td></td>
<td>75%</td>
<td>1891</td>
<td>1395</td>
<td>976</td>
<td>1498</td>
<td>1488</td>
<td>1467</td>
<td>1428</td>
<td>1550</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1710</td>
</tr>
<tr>
<td></td>
<td>Number of FPDs which</td>
<td>8</td>
<td>8</td>
<td>5</td>
<td>7</td>
<td>4</td>
<td>8</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td></td>
<td>survived TCML</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Type of Fracture</td>
<td>4/4</td>
<td>8/0</td>
<td>5/0</td>
<td>2/5</td>
<td>3/1</td>
<td>3/5</td>
<td>1/7</td>
<td>0/8</td>
</tr>
<tr>
<td></td>
<td>veneering/core</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Artificial aging was used to simulate a 5-year period of oral service. The settings were [18]: 1,200,000 mechanical loadings with 50 N and a simultaneous thermal cycling with distilled water between 5°C and 55°C (3,000 times with 2 min each cycle). A human molar was adjusted as antagonist in a three-point contact relation on the pontic of the FPD in an articulator (Amann-Girrbach, Koblach, Austria) and both tooth and FPD were transferred to the simulator. Antagonist-tooth relation was controlled using occlusal foil. All restorations were visually monitored during aging. Failure of the veneering was checked during aging, and the number of loading cycles was recorded. Fractured restorations were excluded from further testing.

After aging, all undamaged FPDs were loaded to failure using a testing machine (Zwick, Ulm, Germany, \( v = 1\) mm/min). The force was applied using a steel ball (\( d = 12 \) mm) while a 1 mm thick tin foil between pontic and antagonist was used to reduce stress concentrations. The FPDs were visually examined before and after fracture testing. Failure mode was classified as fracture of the veneering or the core. Medians, 25\% and 75\% of the fracture resistance [N] were calculated. Statistical analysis was performed using Mann Whitney-U test and Pearson correlation (\( \alpha = 0.05 \)).

### 4.4 Results

The Table 4.1 shows the median fracture results and the number of FPDs which survived TCML. No correlation was found between failure type (core / veneer) and fracture force. TCML reduced the median fracture force from 1735 N (control without TCML #0) to 1227 N (#5), but the difference was not statistically significant (\( p = 0.195 \)). Neither all-ceramic zirconia FPDs (#5) nor porcelain fused to metal (PFM #1 1329 N) reference FPDs showed no significantly different results (\( p = 0.789 \)). Different commercially available ceramic layer materials, Cercon Ceram S 1227 N, Cercon Ceram Kiss 1331 N, Ceramco PFZ 1380 N or Sakura Interaction 1440 N had no significant influence on the fracture force of the FPDs. The fracture force increased with increasing thermal expansion coefficient and firing temperature from 8.7\( \mu \)m/mK / 750°C (#2 533 N), throuth 9.3 \( \mu \)m/mK / 780°C (#4 1098 N) to 9.7 \( \mu \)m/mK /830°C (#5 1227 N). The two materials with firing temperature of 750°C and 760°C had significantly different fracture results with different thermal expansion coefficients of 9.7 \( \mu \)m/mK (#3 1498 N) or of 8.7\( \mu \)m/mK (#2 533 N), respectively. A significant Pearson correlation was found between the fracture force and firing temperature (\( p = 0.062 \)) but not between the fracture force and TEC (\( p = 0.044 \)). No linear regression was found: \( R^2 = 0.731 \) (FT) and \( R^2 = 0.768 \) (TEC) (Fig. 4.1). During TCML chipping was found for group #4 (4 times), group #3 (1x) and group #2 (3x).
Fig. 4.1: Correlation between thermal expansion (TEC), firing temperature (FT) and fracture force.

4.5 Discussion

It has been shown that the loading capacity of ceramic specimens suffers from moisture and dynamical loading. Zirconia-based ceramics lose about 50% of their flexural strength with increasing number of loading cycles [19, 20] and show low-temperature degradation [21]. Similar behaviour is observed for zirconia FPDs: aged with TCML, their fracture resistance is reduced about 30% from 1735 N (#0) to 1227 N (#5). The mechanical degradation over time may be caused either by spontaneous transformation of the tetragonal into monoclinic phase [22, 23] or interactions between core and veneering, but it may also be influenced by processing reactions on the zirconia surface [6, 7, 24]. A further explanation would be a limitation of the partial stabilization. In spite of the TCML-induced degradation, only three out of 64 tested FPDs failed below the assumed chewing force of about 500 N in posterior areas [25]. Most FPDs fractured at a maximum load above 1000 N and thus provided fracture results comparable to PFM FPDs. A bridge construction achieves most of its strength by the core, but a strong influence of the veneer on the fracture potential of the entire construction is shown in finite element analysis [26, 27]. Some authors reported an improvement of the FPD fracture resistance after veneering the core material [5, 28]. The influence of the veneer on the strength of the FPD is underlined by a finite element analysis which showed that constant veneer thickness is requested to achieve optimal, even distribution of the occlusal forces [26]. Bi-layered ceramic systems may be susceptible to chipping due to the combination of a “weak” veneering ceramic with a stronger zirconia core [2]. It has been shown that bi-layered
glass-ceramics deteriorate due to loading in the contact areas. Cracks develop at the contact surface or at the boundary layer between the two ceramics [29-31].

It may be presumed that good bonding between the core and veneering is therefore dependent on the calibration of the two components. In laboratory tests on zirconia, the influence of thermal expansion coefficient (TEC) or transformation processes during sintering has been shown on micro-tensile specimens [9, 32, 33]. Regarding FPDs in TCML, similar findings are noticeable: although the median fracture resistance of the TCML-surviving FPDs was above 1000 N, the variants with 9.3μm/mK /780°C and 9.7μm/mK /760°C showed severe failures of the veneer during TCML. Group #2 provided median loadings of only 533 N until chipping of the veneer. The low fracture resistance of this core-veneering combination may be one explanation for the clinically-reported chipping. The results of this study indicate that the firing temperature for ceramic layering technique of zirconia may not be lower than 830°C combined with a minimal thermal expansion coefficient of 9.2-10.5μm/mK. It may be supposed that the higher firing temperature may cause a better adaptation of the veneering on the core or the low temperature melting glasses may have a better wetting of the zirconia surface. The higher temperature may influence stress concentrations in the bonding, perhaps in combination with a different heating/cooling regime. The smaller TEC of the veneer in comparison to zirconia (10.5μm/mK) results in slight tangential compressive stress, which is supposed to interrupt crack propagation. The results for Sacura Interaction (9.9μm/mK /920°C) or Ceramco PFZ (10.5/900) veneering showed that higher setting of the firing temperature may be effective, too. It is supposed that components that were added for modifying TEC can be excluded for influencing the bonding to the core material directly.

The bonding mechanism between zirconia and veneering glass-ceramics is deficient. The inert zirconia core surface and the glass-ceramic veneering may show only limited chemical adhesion. The bond between two materials is, particularly, a result from differences in the elastic/visco-elastic behaviour between the two layers or, as described, shrinkage of the veneering onto the core material. A small influence of the core surface roughness (due to milling or surface treatment) can be assumed. Extreme roughening of the core on one side may improve the micromechanical interaction, change the surface energy and enhance wetting. On the other side it may damage the core by crack initiation or may reduce the bond to the veneering due to splintering of superficial ceramic layers in the long term. Some investigations showed that sandblasting improved the flexural strength of the zirconia [34] but the enhancement seemed not to be permanent [35]. Beyond this, high pressure or large particle size may damage the zirconia surface or induce crack growth.

Tensile bonding tests mainly showed interfacial failures between the core and veneer [32, 33, 36]. The bond strength between the zirconia and veneer is even reduced to the strength of the
veneer itself [32] and depended on the core treatment, e.g. with liner. It has been shown that
the application of some selenium-based feldspatic porcelain liner/opaquer favourably effects
the bonding [9]. In contrast to the reported interfacial failures of specimens, the fracture
analysis of the tested FPDs showed partly different results. FPDs with high fracture resistance
provided exposed zirconia core, but also thin layers of the veneering on the zirconia surface
(Fig. 4.2).

Fig. 4.2: Three-unit FPD after fracture test: above: chipping of the veneering ceramic; below:
fracture of the core.

Thus, besides the interfacial failure, reverse interpretations of the failure mechanism [8, 27]
may be discussed. Guzatto showed that the tetragonal–monolithic transformation is
accompanied with localized stress, which may consequently result in micro-cracks at the glass
phase of the veneer. This is supposed to lead to a fracture in the veneering layer at a depth of
some hundred micrometers above the core [7]. The damage and sub-critical crack growth in
the long term or with repeated loading may result in chipping – with the result of a thin
veneering layer on the zirconia surface. This may indicate that the bonding between core and
veneering developed to such a high degree that the weak point of the restoration may be shifted towards the strength of the veneering ceramic.

Although in-vitro failures of glass-ceramic FPDs have been investigated [27, 28, 39], no detailed information was provided about zirconia-based FPDs. The clinically available data for zirconia report of 22 adhesively cemented posterior bridges up to one year [2] without any failures. Two chippings were found after one year for 39 conventionally cemented three-unit FPDs and 14 conventionally cemented four-unit FPDs [1] without any further chipping after 18 months [1]. Sailer et al. reported about 11% chipping in 44 FPDs after 42 months [2, 40]. All data are based on the material Cercon Base with Cercon Ceram (#3) or an experimental earlier veneering. The clinical studies coincidently showed no fractures of the high-strength core materials over the whole observation time. Alternative zirconia-based products (Lava; DCZirkon) also show comparable chipping of the veneer [3, 4].

The results suggest that the strength of a zirconia-based FPD might suffer from aging. A correlation between chipping and adapting FT / TEC, as well as the strength of the veneering may be supposed.
4.6 References
