Journal of Oral Rehabilitation

Journal of Oral Rehabilitation 2013 **40**; 51–59

Effect of veneering technique on the fracture resistance of zirconia fixed dental prostheses

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SUMMARY To compare the fracture resistance of zirconia 3-unit posterior fixed dental prostheses (FDPs) frameworks veneered with different veneering materials and techniques before and after artificial ageing. Forty-eight zirconia 3-unit FDPs, representing a missing first molar, were adhesively cemented on human teeth. The zirconia frameworks were randomly distributed according to the veneering materials and techniques into three groups, each containing 16 samples: group LV (layering technique/Vintage ZR), group LZ (layering technique/ZIROX) and group PP (CAD/CAM and press-over techniques/PressXZr). Half of each group was artificially aged through dynamic loading and thermocycling to simulate 5 years of clinical service. Afterwards, all specimens were tested for fracture resistance using compressive load. An analysis of variance (ANOVA) was used to assess the effect of veneering ceramic and artificial ageing on fracture resistance ($P < 0{\cdot}05$). Except for one minor cohesive chipping in group LV1, all specimens survived artificial ageing. The mean fracture resistance values (in Newton) of different non-aged (± s.d.)/aged (± s.d.) groups were as follows: LV0 2034 (± 401)/LV1 1625 (± 291); LZ0 2373 (± 718)/LZ1 1769 (± 136); and PP0 1959 (± 453)/PP1 1897 (± 329). Artificial ageing significantly reduced the fracture resistance in groups veneered with the layering technique ($P < 0{\cdot}05$), whereas no significant effect was found in specimens veneered with the CAD/CAM and press-over techniques. All tested systems have the potential to withstand occlusal forces applied in the posterior region. The combination of the CAD/CAM and press-over techniques for the veneering process improved the overall stability after artificial ageing, relative to the layering technique.

KEYWORDS: zirconia, fixed dental prostheses, layering, computer-aided design/computer-aided manufacturing, press-over

Accepted for publication 24 April 2012

Introduction

Compared to metal alloys, zirconia ceramics offer very good aesthetics, excellent biocompatibility, chemical and dimensional stability, low plaque accumulation, low thermal conductivity, low radioactivity and high radioopacity (1–6). The mechanical properties of zirconia are the best among dental ceramics, with a flexural strength of 900–1200 MPa and fracture toughness of 9–10 mN/m$^{3/2}$ (7). The high strength of the material can be explained by the phase transformation toughening mechanism, which relies on a crystal structural change (tetragonal to monoclinic) under stress to inhibit the propagation of an advancing crack (3). However, the material has been reported to undergo an irreversible slow tetragonal to monoclinic transformation known as low-temperature degradation (LTD) which, in turn, can cause mechanical property degradation (8).

Y-TZP-based restorations are usually manufactured through computer-aided design/computer-aided manufacturing (CAD/CAM) technology. An enlarged framework is designed and milled from homogenous soft blanks of zirconia, which are usually delivered in non-sintered or pre-sintered stages. The framework structure has a linear shrinkage of 20–25% during sintering until it reaches the final dimensions (9, 10).

 doi: 10.1111/j.1365-2842.2012.02323.x

Zirconia frameworks are usually veneered manually by means of the layering technique. In an effort towards entirely digitising production, including the veneering process, CAD/CAM technology can be employed to mill a resin replica of the veneering porcelain. The milled resin replicas serve as moulds for the press-over technology (11, 12), in which the veneering ceramic pellets are heated and pressed into the investment mould directly over the framework. Alternatively, the sintering technique (CAD-on technique) has been developed to veneer zirconia frameworks with corresponding CAD/CAM-fabricated lithium-disilicate glass–ceramic veneering copings (13). The two components (CAD/CAM framework and CAD/CAM veneer) are joined by means of a low-fusing ceramic material or adhesive cementation procedures. Both novel veneering techniques aim to overcome human performance inconsistencies, as well as to improve quality, reliability and cost-effectiveness of CAD-/CAM-fabricated zirconia-based restorations.

A limited number of short-term and mid-term clinical studies showed promising results of Y-TZP ceramic for fixed dental prostheses (FDPs) frameworks in the posterior region (14–24). Nevertheless, despite different modifications in fabrication protocols and materials, chipping of the veneering material is still considered a major drawback of zirconia-based restorations (25, 26).

Moreover, it is unknown whether newly introduced veneering techniques will minimise or eliminate the chipping problem. An investigation of the effects of the different veneering techniques on the stability of zirconia-based restorations under clinically relevant conditions would provide useful information and guidelines about the applicability of these techniques. Therefore, the aim of this *in vitro* study was to compare the fracture resistance of zirconia 3-unit posterior FDP frameworks veneered with various veneering materials and techniques before and after artificial ageing.

Material and method

Forty-eight caries-free human mandibular second premolars and 48 second molars were selected and stored in a 0·1% thymol solution throughout the study. Pairs of teeth were randomly assigned and imbedded in a self-curing modified polyester resin*, so as to create 48 clinical models representing a missing first molar (span length of 11 mm). To simulate the periodontal ligament (PDL), the roots were covered 2 mm apically from the cemento-enamel junction (CEJ) with a thin layer of a gum resin (0·25 mm)†. Circumferential chamfer of 0·8 mm in depth, a convergence angle of 10° and a wall height of 5 mm for both teeth were prepared (Fig. 1). Afterwards, impressions using polyvinyl siloxane were made‡ and working dies were poured using type 4 stone§. The stone dies were scanned using a 3D-laser scanner¶, to enable framework design using CAD software (3Shape DentalDesigner¶). The design settings for all frameworks were standardised as follows: the minimal wall thickness was 0·6 mm and the minimal cross-sections were 10 and 12 mm^2 for the mesial and distal connectors, respectively. Next, the frameworks were milled from pre-sintered Y-TZP blanks (ZENOTEC Zr Bridge¶) in a milling machine (ZENOTE 4030 M1¶). The machine mills an enlarged framework to compensate for the later sintering shrinkage. The frameworks were then sintered at a temperature of 1580 °C for 4 h in a sintering furnace (ZENOTEC Fire¶). The coefficient of thermal expansion (CTE) of sintered zirconia is $10{\cdot}5 \times 10^{-6}$ K^{-1}. Subsequently, the hard-sintered frameworks were checked for internal and marginal fit and adjusted if necessary using water-cooled diamond burs** under 4·5× magnification.

All frameworks were randomly assigned to various veneering materials and techniques (three groups of 16 samples each). The first group was veneered using the layering technique with leucite-strengthened feldspathic porcelain†† (group LV), with a CTE of $9{\cdot}4 \times 10^{-6}$ K^{-1}. The second group was veneered using the layering technique with leucite-free high-density advanced microstructure (HDAM) feldspathic porcelain (ZIROX¶). (group LZ), with a CTE of 10×10^{-6} K^{-1}. Four firings were required for each sample of the both first and second groups (liner, dentin + enamel 1, dentin + enamel 2 and glazing). The layering process was performed by the same dental technician, with the goal of obtaining a homogenous veneering thickness of a range between 0·6 mm at margins and 1·5 mm at occlusal surfaces.

*Technovit 4000; Kulzer, Wehrheim, Germany.

†Anti-Rutsch-Lack; Wenko-Wenselaar GmbH, Hilden, Germany.

‡Dimension Garant L and Firmer Set Putty; 3M-Espe, Seefeld, Germany.

§Fujirock type 4; GC, Tokyo, Japan.

¶3shape D 700; Wieland, Pforzheim, Germany.

**No ZR8850; Gebr. Brasseler, Lemgo, Germany.

††VINTAGE ZR; Shofu Dental GmbH, Ratingen, Germany.

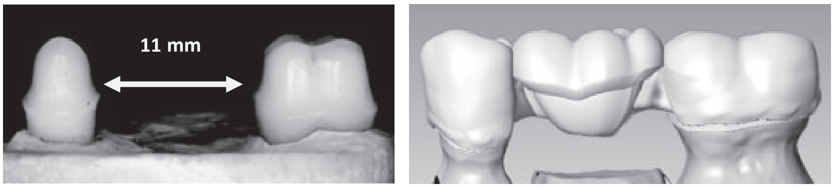

Fig. 1. Dimensions of the test model used with the prepared teeth (left); a representative image showing the standardised design of a fixed dental prostheses from CAD/press-over group, in which both framework and veneering are CAD-designed (right).

The third group was veneered using the CAD/CAM and press-over techniques with a leucite-free high-density advanced microstructure (HDAM) feldspathic porcelain (PressXZr[¶]). (group PP), with a CTE of $9{\cdot}3 \times 10^{-6}\ K^{-1}$. The same CAD software was used to digitally construct the veneering material, with a thickness range from minimally 0·6 mm at the margins to maximally 1·5 mm at the occlusal surfaces (Fig. 1). Resin replicas were milled from castable PMMA blanks (ZENOTEC PMMA cast[¶]) and then attached to the frameworks with a casting wax (Creative casting wax[¶]) (Fig. 2). Each framework was attached to a 200-g muffle ring (PressXMuffel[¶]). The rings were filled with a vacuum-mixed investment material (PressXZr Investment Ref. No. 87-95-00-2500[¶]), and after a setting time of 45 min, the rings were pre-heated for 60 min at 900 °C to burn out the wax and resin parts entirely. Ceramic pellets of A2 shade (PressXZr pellet staining[¶]). were then pressed in the designated press furnace (DEKEMA Austromat 3001 press-i-dent[¶]). The muffles were allowed to cool slowly to room temperature for at least 30 min. Following this, the bulks of the investment were removed carefully, first with a diamond disc and then by blasting with 50-μm grain size resin beads at 1 bar. Subsequently, the spures were separated and the veneering ceramic was contoured and finished. Finally, the pressed FDPs were carefully cleaned using a steam cleaner unit[‡‡], followed by a glaze firing (PressXZr Glaze[¶]).

Airborne particle abrasion was applied to the inner surface of the frameworks with 50-μm Al_2O_3 particles[§§] at an air pressure of 2·5 bar for 10–15 s (27, 28). Then, the FDPs were ultrasonically cleaned in 96% isopropyl alcohol. After drying, ceramic primer was applied to the inner surface of the FDPs[¶¶]. The teeth were cleaned with a rubber cup using a fluoride-free prophylaxis paste[***]. Afterwards, the FDPs were adhesively cemented using self-adhesive resin cement under finger pressure[†††] (29). All FDPs were stored in water for a minimum of 7 days prior to mechanical testing to allow hydration of the resin cement (30, 31).

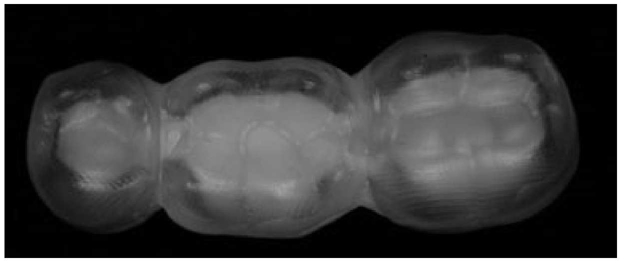

Fig. 2. Example of a CAD-/CAM-generated PMMA replica of the designed veneering layer from CAD/press-over group. The replica is adapted over the framework and ready for the investing process.

Half of each group ($n = 8$) was artificially aged by exposure to 1 200 000 cycles of thermo-mechanical fatigue in a computer-controlled dual-axis artificial oral environment[‡‡‡]. Thus, a total of six subgroups were formed, labelled as follows: LV0, LV1, LZ0, LZ1, PP0 and PP1 (0 – non-aged; 1 – aged). Using a 6-mm-diameter ceramic ball (Steatite, Hoechst Ceram Tec, Wunsiedel, Germany), a 49-N load was applied on the centre of the occlusal surface of each pontic at a frequency of 1·6 Hz. During testing, all specimens were subjected to simultaneous thermal cycling between 5 and 55 °C for 60 s each, with a dwell time of 12 s. Finally, all aged and non-aged specimens were loaded compressively in a universal testing machine[§§§] with force application perpendicular to the occlusal surface and a cross-head speed of 2 mm/min. The loading

[‡‡]DENTAL 2001, JAEGER, Wiernsheim, Germany.
[§§]Cobra 50 μm white; Renfert GmbH, Hilzingen, Germany.
[¶¶]Clearfil Ceramic Primer; Kuraray Medical, Osaka, Japan.

[***]NUPRO; DENTSPLY Detrey, Konstanz, Germany.
[†††]RelyXUnicem; 3M ESPE, Seefeld, Germany.
[‡‡‡]Chewing Simulator CS-4 professional line; SD Mechatronik GmbH, Feldkirchen-Westerham, Germany.
[§§§]Z010/TN2S; Zwick, Ulm, Germany.

Table 1. Values of fracture resistance test for different groups in Newton to be used for the LSMEANS procedure for ANOVA

Group	Ageing	No. obs.	Mean value	s.d.	Minimum value	Lower quartile	Median value	Upper quartile	Maximum value
LV	0	8	2034	± 401	1632	1725	1979	2194	2846
	1	8	1625	± 291	1172	1413	1673	1849	1959
LZ	0	8	2373	± 718	1353	1812	2395	2993	3234
	1	8	1769	± 136	1597	1664	1730	1912	1947
PP	0	8	1959	± 453	1210	1618	2086	2275	2507
	1	8	1897	± 329	1567	1659	1807	2068	2538

No. obs., number of observations; s.d., standard deviation; group LV, layering technique/Vintage ZR; group LZ, layering technique/ZIROX; Group PP, CAD/CAM and press-over techniques/PressXZr; 0, non-aged groups; 1, aged groups.

stamp was centrally positioned over the occlusal surface of the first molar (pontic). A one-millimetre-thick tin foil[¶¶¶] was placed between the loading stamp and the pontic to achieve homogenous stress distribution and to avoid force peaks. The load required to fracture the specimen was recorded using x-t recording software[****].

Results of the load-to-fracture test were presented with the help of box plots. An analysis of variance (ANOVA) was used to assess the effect of veneering ceramic and artificial ageing on the fracture resistance (FR). Least-square means (LSMEANS) and pairwise differences of LSMEANS were calculated, and P values were adjusted by the Tukey–Kramer method to a global significance level of $P < 0{\cdot}05$.

Results

Except for one minor cohesive chipping in group LV1, which occurred after 1 000 000 cycles, all aged FDPs survived artificial ageing without signs of chipping or decementation. The mean fracture resistance values (in Newton) of different non-aged (±s.d.)/aged (±s.d.) groups were as follows: group LV0 2034 (±401)/group LV1 1625 (±291); group LZ0 2373 (±718)/group LZ1 1769 (±136); and group PP0 1959 (±453)/group PP1 1897 (±329) (Table 1).

The smallest single-sample failure load (1172 N) was observed in the LV1 group, whereas the highest single-sample value (3234 N) was observed in the LV0 group. The highest mean failure load value in non-aged groups was observed in the LZ group, followed by the LV group and the PP group. In aged groups, the highest mean failure load value was recorded for the PP group, followed by LZ group and the LV group.

Compared to values for groups without artificial ageing, artificial ageing reduced the mean fracture resistance by 20%, 25% and 4% for groups LV, LZ and PP, respectively. Hence, artificial ageing significantly reduced the fracture resistance in groups with layering technique (LV and LZ) ($P < 0{\cdot}05$), whereas no significant effect was found in groups veneered with the CAD/CAM and press-over techniques (PP) ($P > 0{\cdot}05$) (Fig. 3).

All investigated FDPs failed with a sudden bulk fracture using the loading test, except for one sample

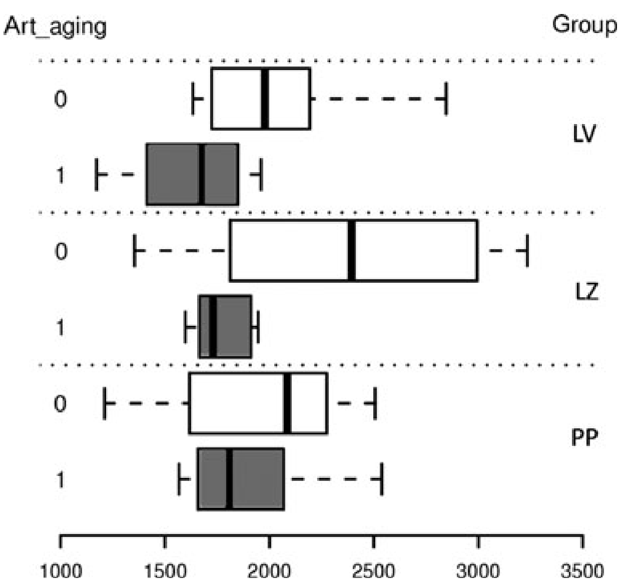

Fig. 3. Box plots of results of the load-to-fracture test of different groups in Newton (group LV, layering technique/Vintage ZR; group LZ, layering technique/ZIROX; Group PP, CAD/CAM and press-over techniques/PressXZr) before and after ageing (0, non-aged groups; 1, aged groups) ($n = 8$).

[¶¶¶]Dentaurum, Ispringen, Germany.

[****]ZwicktestXpert V7.1; Zwick, Ulm, Germany.

from the LV1 group, which exhibited a catastrophic chipping without any fracture of the framework. The fracture patterns of the test specimens of all groups were located in the loading point going through one of the connectors, while in four samples, the fracture involved both connectors.

Discussion

The aim of this *in vitro* study was to evaluate the fracture resistance of zirconia 3-unit posterior FDPs veneered with various veneering materials and techniques, before and after artificial ageing. For many years, conventional 3-unit FDP was considered to be the best treatment option for the replacement of a missing posterior single tooth. To date, implant-supported single crown has become a popular alternative for posterior single-tooth replacement (32). This treatment modality saves adjacent teeth from treatment, but the operative procedure is extensive. Decision making should be based on clinical and radiographic assessments as well as on the knowledge of the long-term survival and complication rates of each of these therapeutic options (33).

In the absence of randomised clinical trials, carefully designed *in vitro* studies that closely simulate clinical situations are important sources of information regarding the potential longevity and possible complications of a given treatment modality (34). The current study design, materials used and the selected simulation device were carefully selected to closely imitate clinical conditions. Extracted human teeth were used because their characteristics are closer to the clinical situation than those of metal, resin or animal teeth (35). Moreover, abutment mobility in the current study was insured through the application of a thin layer of gum resin, with the aim to simulate physiological tooth movement (36). With this layer, the artificial tooth mobility under a force of 5 N has been reported to be 100 ± 30 μm in the horizontal direction and 65 ± 21 μm in the vertical direction (37). These values are similar to the physiological tooth mobility described in the literature (38). Nevertheless, owing to its complex structure (collagen fibre network, blood vessels, nerves, fluids, etc.) (39), the biomechanics of the natural PDL are highly elaborated to simulate (38).

In this study, half of the specimens of each test group were exposed to an artificial oral environment. The parameters used for cyclic loading were limited to physiological values (40, 41): a cyclic loading force of 49 N was applied so as to approach a clinically relevant situation, as described in several similar *in vitro* studies (35, 42–44). These studies have considered the functional forces that arise during mastication or swallowing, which usually range between 2 and 50 N (40, 41). The three-dimensional masticatory load curve is programmed by the combination of the horizontal (0·5 mm) and the vertical (6 mm) motion, resulting in a precisely defined vertical impact and horizontal sliding under contact. Nevertheless, the loading was restricted to the centre of the occlusal surface of each pontic.

Dynamic loading was combined with thermocycling to simulate ageing of the specimens. The rapid change in temperature when the FDPs are submerged in baths could create stresses in the specimens between the surface and the bulk material. When tension and compression periodically occur at a crack tip as a result of load cycles, the damage is increased by access to water. Hence, small defects introduced during the fabrication process may have a detrimental effect on the fatigue life of zirconia FDPs. Studies have shown that humans have an average of 250 000 masticatory cycles per year. Comparing several *in vitro* studies, the number of applied cycles varies according to the selected service time (42, 43, 45). In the present study, the specimens were subjected to 1 200 000 masticatory cycles to simulate a service time of 5 years, as previously reported (35, 42–45).

It should be mentioned, however, that the number of specimens tested, the use of water rather than artificial saliva during testing, the difficulty in simulating the sensory regulated tooth mobility and natural ageing in their nature and frequency, as well as the location of the loading point, are limitations of this study that may influence the clinical interpretation of the results.

It has been shown that masticatory loads range from 50 to 250 N, while parafunctional behaviours such as bruxism can produce loads between 500 and 1000 N (46–52). In this study, all test groups before and after artificial ageing showed minimum fracture resistance levels greater than 1170 N. Thus, all test specimens exceeded the limits of fracture resistance for posterior restorations, as confirmed by previous *in vitro* studies (42, 44, 53). Nevertheless, the materials and techniques used in other reports are quite different than those used in the present study, making a direct comparison rather difficult.

Except for one minor cohesive chipping in group LV1, no other chippings were reported after exposure to artificial ageing. Because a single occurrence is sporadic and not statistically significant in our data set, the issue was not further investigated. Clinical studies have reported chipping rates for FDPs veneered with the layering technique of approximately 7·5–25% after an observation period between 3 and 5 years (14, 16, 19, 22, 54). Cohesive veneer fractures have also been reported in zirconia-based FDPs veneered using the press-over technique (17). This cohesive failure pattern of the veneering material suggests a sufficient interfacial bond between the two systems, which has been confirmed *in vitro* (55). Residual thermal stresses have been recently suggested as a cause for the prevalence of veneered zirconia-based restoration chipping (56). These residual stresses may result from the mismatch of the larger CTE of the veneering ceramic (57), thickness of the veneering porcelain, number of firings and/or the cooling rate. In particular, a fast cooling rate could create major thermal gradients within the porcelain, an attribute that is directly related to the low thermal conductivity of zirconia. Therefore, slow cooling of the restoration above the glass transition temperature of the porcelain is highly recommended to reduce the incidence of chipping (58). Moreover, microstructural defects in the veneering porcelain, including porosities, agglomerates and large grain zones, are other possible factors that could promote chipping of the veneering ceramic (59). In an *in vitro* study, investigators observed higher microtensile bond strength of zirconia specimens veneered with CAD/-CAM/press-over technique compared to manually veneered specimens. The specimens veneered with CAD/CAM/press-over technique demonstrated sufficient contact between the two materials, whereas the manually layered zirconia interface showed structural defects and air bubbles (11).

The application of artificial ageing in this study caused a significant decrease in mean fracture strength: approximately 20–24% for the groups LV1 and LZ1, which were veneered using the layering technique. However, no significant decrease in mean fracture strength was recorded for group PP, which was veneered with the press-over technique. Accordingly, the type of veneering process cannot be excluded as a factor that may affect the fracture resistance of Y-TZP-based FDPs. These interesting findings suggest a hypothesis. When comparing the press-over and layering techniques, the latter technique is usually associated with a significant exposure of the zirconia frameworks to moisture and several 'heating' steps during the firing procedure. Exposure to moisture and heat are known to trigger LTD in the zirconia surface (8, 60). In fact, a 20-min exposure of zirconia to 132 °C in an autoclave has been demonstrated to induce significant material surface ageing, equal to 5 years of loading (61). Conversely, the press-over technique is not associated with any exposure to moisture and requires a reduced number of heating steps compared to the layering technique. Therefore, it can be assumed that the layering technique induces more LTD in the framework's surface than the press-over technique, thus reducing surface stability. Accordingly, the stability of the overlying veneering ceramic layer may be reduced. In addition, a smaller number of firing steps for specimens veneered with the press-over technique may reduce residual thermal stresses and deformation (56). Furthermore, press-over technology would improve homogeneity and density of the veneering material, with fewer voids within the bulk of the veneering material and at the interface. Consequently, the mechanical strength of the veneering material may be improved because of a more equal distribution of the strain within the veneering ceramic. As no information is available in the literature, further studies are needed to validate the proposed hypothesis.

All investigated FDPs failed with a sudden bulk fracture using static loading test, except for one FDP from the LV1 group, which exhibited catastrophic chipping without any fracture involving the framework. The fracture patterns of the test specimens of all groups included both the loading point and (at least) one of the connectors. In four FDPs, the fracture involved both connectors. These patterns are similar to those previously reported *in vitro* for 3-unit zirconia-based all-ceramic FDPs (42, 45, 53, 62). It was difficult to assess the exact origin of the fractures that occurred in the present study, that is, whether they started at the loading point or at the connectors. However, they were perpendicular to the mesial/distal axis of the frameworks in a smooth curve between the loading point and the gingival side of the connector.

In addition, the dimensions of the connector area are crucial for the resistance and longevity of all-ceramic FDPs (63). A small, irregularly designed connector area will reach the critical strain faster than a thick, well-designed connector. In the current study, connector dimensions of 10 mm^2 for the mesial and 12 mm^2 for the distal connector were used to simulate the clinical condition. Several *in vitro* studies investigating the fracture resistance of Y-TZP-based all-ceramic FPDs showed appropriate results with connector dimensions of 9 mm^2 (53, 63).

Conclusion

Within the limits of this *in vitro* study, all tested Y-TZP-based FDPs have the potential to withstand potential occlusal forces applied in the posterior region. The combination of the CAD/CAM and the press-over techniques for the veneering process improves the stability after artificial ageing relative to the layering technique. Before they can be recommended for daily application, the effect of newly introduced veneering techniques on the long-term stability of Y-TZP-based zirconia FDPs must be verified in well-designed, randomised clinical trials.

Acknowledgments

The authors would like to acknowledge Wieland, Pforzheim, Germany, for the technical assistance with the fabrication of zirconia frameworks, in addition to the veneering of the third group using the CAD/CAM and press-over techniques.

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