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Flexural strength and failure modes of layered ceramic structures

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Abstract

Objective—to evaluate the effect of the specimen design on the flexural strength (σ_f) and failure mode of ceramic structures, testing the hypothesis that the ceramic material under tension controls the mechanical performance of the structure.

Methods—Three ceramics used as framework materials for fixed partial dentures (YZ - Vita In-Ceram YZ; IZ - Vita In-Ceram Zirconia; AL - Vita In-Ceram AL) and two veneering porcelains (VM7 and VM9) were studied. Bar-shaped specimens were produced in three different designs (n=10): monolithic, two layers (porcelain-framework) and three layers (TRI) (porcelain-framework-porcelain). Specimens were tested for three-point flexural strength at 1 MPa/s in 37°C artificial saliva. For bi-layered design, the specimens were tested in both conditions: with porcelain (PT) or framework ceramic (FT) layer under tension. Fracture surfaces were analyzed using stereomicroscope and scanning electron microscopy (SEM). Young's modulus (E) and Poisson's ratio (ν) were determined using ultrasonic pulse-echo method. Results were statistically analyzed by Kruskal-Wallis and Student-Newman-Keuls tests.

Results—Except for VM7 and VM9, significant differences were observed for E values among the materials. YZ showed the highest ν value followed by IZ and AL. YZ presented the highest σ_f . There was no statistical difference in the σ_f value between IZ and IZ-FT and between AL and AL-FT. σ_f values for YZ-PT, IZ-PT, IZ-TRI, AL-PT, AL-TRI were similar to the results obtained for VM7 and VM9. Two types of fracture mode were identified: total and partial failure.

Significance—The mechanical performance of the specimens was determined by the material under tension during testing, confirming the study hypothesis.

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Keywords

dental ceramics; monolithic and multilayer structures; strength; fractography; fracture surface

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Introduction

The use of all-ceramic restorations has increased since the introduction of CAD-CAM technology in dentistry. Ceramic systems with high crystalline content have good fracture resistance but are highly opaque. Therefore, to obtain a natural-looking restoration the framework material should be veneered with porcelain, which presents better esthetics [1, 2]. Usually the mechanical behavior of veneer and framework materials is evaluated separately, providing important information on the fracture resistance of each component of the restoration. However, it is also important to understand how these materials interact in a multi-layer configuration. The study of layered structures can provide valuable information about the stress distribution, failure mode and origin, interfacial bonding and thermal stresses [2, 3].

Interfaces can have significant influence on the mechanical performance of layered structures. For instance, the slow crack growth behavior can be affected by the interface fracture energy and the difference between the elastic modulus of the coupling materials [4–7]. In addition, a mismatch in the coefficient of thermal expansion (CTE) between the ceramics produces residual stresses near the interface that could influence the fracture mode of these laminates [8–12]. Other factors that are also related to the performance of these structures are the mechanical properties of the material subjected to maximum tensile stress during loading and the thickness ratio between layers [2, 5, 7, 13–16]. Hsueh et al. (2008) [17] used finite element analysis (FEA) and reported that the location of the maximum tensile stress changes with the thickness ratio between the veneering and framework materials.

Investigations showed that the material subjected to tension during flexural testing and the interaction between the materials in ceramic structures have a significant effect in the strength and fracture mode of layered structures [7, 14, 16, 18]. When the framework material is under tension, in layered structures, the fracture strength values tend to be similar to the ones obtained by the monolithic specimens made of framework material [7, 19]. On the other side, the framework material has a small influence in the fracture strength of these structures when the porcelain is subjected to tension [20].

Multi-layered structures are also subjected to residual stresses from mismatches in the CTE between the materials and thermal gradients produced during cooling. The restoration veneering process involves high temperature sintering (750–900°C) and subsequently cooling to room temperature. Usually, in both metal-ceramic and all-ceramic restorations, the porcelain CTE is slightly lower than the framework CTE, producing compressive stresses in the porcelain surface and compensatory tensile stresses in the framework surface [2, 8, 9, 21]. However, for all-ceramic restorations, the framework material is brittle and the risk of destructive stresses in the veneering layer is higher than in metal-ceramic restorations [22]. Another limitation of all-ceramic restoration is the fact that structures produced with porcelain combined to a material with low thermal diffusivity, such as yttria partially stabilized zirconia ceramic (YZ), are more susceptible to the development of tensile stresses [2, 10]. These residual stresses are induced by the large temperature difference between layers during cooling and may result in cracks and porcelain chipping [2, 8, 10].

Although bi-layered structures have been studied in terms of fracture strength, failure mode and stress distribution, there are no studies evaluating tri-layered structures (porcelain/framework/porcelain), which is the real configuration of a FPD connector. Therefore, the objective of this study was to systematically evaluate the effect of the specimen design on the flexural strength and failure mode of ceramic materials, testing the hypothesis that the material under tension during testing influences the flexural strength values and failure behavior of these structures. In addition, the mechanical behavior of different materials used to produce all-ceramic FPDs was characterized.

Materials and Methods

Three ceramics used as framework materials for FPDs and two veneering porcelains were studied. The materials used in this study are described in Table 1. Three specimen designs were produced with the dimensions of 2 mm x 4 mm x 16 mm:

1. monolithic (one material);
2. two layers, 1 mm thickness of framework ceramic and 1 mm thickness of porcelain;
3. three layers, 1 mm thickness of framework ceramic completely veneered with 0.5 mm thickness of porcelain.

YZ, IZ and AL bar-shaped specimens were obtained by cutting pre-sintered blocks using a diamond disc in a precision cutting machine (Isomet 1000, Buehler, Lake Bluff, USA) at 275 rpm. YZ and AL specimens were sintered in the Zyrcomat furnace (Vita Zahnfabrik, Germany). IZ material was infiltrated with glass (Zirconia Glass Powder, Vita Zahnfabrik, Germany). The infiltration cycle was performed in the Inceramat 3 furnace (Vita Zahnfabrik, Germany), and the excess glass was removed with burs. The glass infiltration cycle was performed at 1110°C for 6 hours, according to the manufacturer's instruction. VM7 and VM9 specimens were fabricated by mixing ceramic powder with distilled water to form a slurry that was poured into a metallic mold and condensed with manual vibration. A Keramat I furnace (Knebel, Porto Alegre, Brazil) was used to perform the porcelain sintering. The porcelain specimens were sintered according to the following cycle: pre-drying at 500°C for 6 min, heating to 910°C at a rate of 55°C/min under vacuum, heating at 960°C for 1 min and cooled down to room temperature (6 min).

After YZ, AL, VM7 and VM9 sintering and IZ glass infiltration, monolithic specimens were ground to their final dimensions (2 mm x 4 mm x 16 mm), and the 4-mm wide face was polished to a 1 µm finish using a polishing machine (Ecomet 2, Buehler, Lake Bluff, USA). All edges were chamfered at a 0.1 mm wide chamfer, as proposed by ISO 6872:2008 [23].

For multi-layer structures (two- and three-layer specimens), the framework material was veneered with the porcelain recommended by the manufacturer: VM7 was used for IZ and AL, and VM9 was used for YZ. Framework bars were produced following the methodology described for monolithic specimens. For two-layer structures, the framework material (1 mm x 4 mm x 16 mm) was veneered with a 1 mm thickness of porcelain using a metallic mold. After sintering, half of the specimens (n=10) had the framework surface polished, and the other half had the porcelain surface polished. For tri-layered structures, framework ceramic bars with the dimensions of 1 mm x 3 mm x 15 mm were produced and all surfaces were veneered with a 0.5 mm thickness of porcelain. One of the 4-mm wide faces was also polished to a 1 µm finish. After polishing, all edges were chamfered. For YZ material, before veneering, a bonding agent (Effect Bonder, Vita Zahnfabrik, Germany) was applied to the specimen surfaces and sintered according to the manufacturer's instructions.

Specimens were classified according to the number of layers (one, two or three layers) and to the flexural test configuration (porcelain or framework in tension) (Table 2). Therefore, this study tested 14 experimental groups (n=10) for flexural strength.

The mechanical testing was performed according to the ISO 6872:2008 with a three-point flexure fixture with 2-mm diameter rollers and 12.0 mm span. The fixture was immersed in 37°C artificial saliva with the following composition: 100 mL of KH_2PO_4 (2.5 mM); 100 mL of Na_2HPO_4 (2.4 mM); 100 mL of KHCO_3 (1.50 mM); 100 mL of NaCl (1.0 mM); 100 mL of MgCl_2 (0.15 mM); 100 mL of CaCl_2 (1.5 mM); and 6 mL of citric acid (0.002 mM) [24]. The flexural strength (σ_f) was determined using a universal testing machine (Sintech 5G, MTS, São Paulo, Brazil) at a constant stress rate of 1 MPa/s.

The maximum tensile stress can be calculated using the following the equation [7, 25]:

$$\sigma_f = \frac{MY}{I} \quad (1)$$

where M is the moment of the load, Y is the distance from the neutral axis to the outermost fiber, and I is the moment of inertia of the cross-section about the central axis.

For the three-point flexural strength test of monolithic specimens: $M=(PL)/4$, $Y=t/2$, and $I=(1/12)wt^3$. By substituting into equation 1, the following equation is developed [23]:

$$\sigma_f = \frac{3PL}{2wt^2} \quad (2)$$

where, P is the fracture load (N), L is the span (12 mm), w is the specimen width (mm), and t is the specimen thickness (mm).

For bi-layered specimens the σ_f was determined by the equation [7, 25]:

$$\sigma_f = \frac{6M}{wt_t^2 K} \left(2 + \frac{t_c}{t_t} + \frac{E_t t_t}{E_c t_c} \right) \quad (3)$$

where K is calculated according to:

$$K = 4 + 6 \frac{t_c}{t_t} + 4 \left(\frac{t_c}{t_t} \right)^2 + \frac{E_c}{E_t} \left(\frac{t_c}{t_t} \right)^3 + \frac{E_t t_t}{E_c t_c} \quad (4)$$

Replacing M and K (equation 4) in equation 3, the equation 5 is used to calculate the flexural strength (σ_f):

$$\sigma_f = \frac{3E_t L P (E_c t_c^2 + 2E_c t_c t_t + E_t t_t^2)}{2w (E_c^2 t_c^4 + 4E_c E_t t_c^3 t_t + 6E_c E_t t_c^2 t_t^2 + 4E_c E_t t_c t_t^3 + E_t^2 t_t^4)} \quad (5)$$

For tri-layered structures equation 1 still applies but new values of Y and I (Y' and I^{TOT}) need to be calculated as follows [7, 25]:

$$Y' = \frac{\left[\left(\frac{t_c^2}{2} \right) + \frac{E_f}{E_t} \left[\left(\frac{t_f^2}{2} \right) + t_f t_c \right] + \frac{E_c}{E_t} \left[\left(\frac{t_c^2}{2} \right) + t_c t_f + t_c t_t \right] \right]}{\left[t_t + \left(\frac{E_f}{E_t} \right) t_f + \left(\frac{E_c}{E_t} \right) t_c \right]} \quad (6)$$

$$I^{TOT} = \left\{ \left[\left(\frac{1}{12} \right) \left(\frac{E_c}{E_t} \right) w t_c^3 \right] + \left[\left(\frac{E_c}{E_t} \right) w t_c \left[t_t + t_f + \left(\frac{t_c}{2} \right) - Y' \right]^2 \right] + \left[\left(\frac{1}{12} \right) \left(\frac{E_f}{E_t} \right) w t_f^3 \right] + \left[\left(\frac{E_f}{E_t} \right) w t_f \left[t_t + \left(\frac{t_f}{2} \right) - Y' \right]^2 \right] + \left[\left(\frac{1}{12} \right) w t_t^3 \right] + \left[w t_t \left[\left(\frac{t_c}{2} \right) - Y' \right]^2 \right] \right\} \quad (7)$$

In equations 3–7, t_t , t_c and t_f are the thickness (mm) of the materials under tension, compression and framework, respectively; and E_t , E_c and E_f are the Young's modulus (GPa) of the materials under tension, compression and framework, respectively; w is the width (mm); P is the load (N) and L is the span (12 mm). The thickness of the materials in the multi-layered structures was measured using an optical microscope after specimens fracture.

Fracture surfaces were examined using stereomicroscope (Olympus SZ61, Olympus, Japan) and scanning electron microscope (SEM) (Stereoscan 440, LEO Electron Microscopy Ltd, Cambridge, England) to determine the mode of failure based on the fracture origin and fractographic principles [26].

The polished porcelain surface of specimens from groups VM9, YZ-PT and YZ-TRI were indented using a Vickers micro-hardness tester (load: 2 kg; dwell time: 20 s). The residual stress (MPa) was calculated according to the following equation:

$$\sigma_r = \frac{K_{Ic} - K_{Ic'}}{2 \sqrt{\frac{c}{\pi}}} \times 1000 \quad (8)$$

where, K_{Ic} is the mean fracture toughness of VM9, $K_{Ic'}$ is the mean fracture toughness of YZ-PT or YZ-TRI groups, and c is half size of the median crack.

Young's modulus (E) and Poisson's ratio (ν) values were obtained by the pulse-echo technique (pulser-receiver 5900 PR, Panametrics, Waltham, MA, USA) according to ASTM E 494–95 standard [27]. Density (ρ) was determined using the Archimedes principle [2].

Data for σ_f were analyzed using Kruskal-Wallis one-way analysis of variance on ranks and Student-Newman-Keuls method for multiple comparisons. One-way analysis of variance and Tukey test with a significance level of 5% was used for E , ν and ρ results.

Results

Table 3 shows the mean and standard deviation values of density (ρ), Young's modulus (E) and Poisson's ratio (ν) for the studied materials. Significant differences were found among mean ρ values. YZ showed the greatest mean ρ value, followed by IZ, AL, VM9 and VM7. Significant differences were found for the mean E values with AL showing the greatest value and the porcelains (VM7 and VM9) the lowest values. Considering the ν data, YZ showed the highest mean value.

Table 4 shows the median and standard deviation values of flexural strength (σ_f) for the experimental groups. YZ showed the highest median σ_f value. There was no statistical difference in the median σ_f values between IZ and IZ-FT and between AL and AL-FT. The

median σ_f values for YZ-PT, IZ-PT, IZ-TRI, AL-PT, AL-TRI groups were not statistically different from the median σ_f values found for porcelain monolithic groups (VM7 and VM9). However, the median σ_f value for YZ-TRI group was higher than the median σ_f values found for YZ-PT and monolithic porcelain VM9 group.

After visual inspection of the specimens broken in flexural test, two types of failure modes were identified: total fracture and partial failure (fracture of only one layer). All specimens with two layers tested with the porcelain under tension had partial failure. In these cases, the initial flaw originated in the porcelain surface, propagated through the porcelain layer and deflected near the interface, before reaching the framework material (Figure 1). The mechanical test was interrupted when the porcelain layer failed, and the maximum load obtained was used to calculate the flexural strength (σ_f).

All bi-layered specimens from IZ and AL materials tested with framework material in tension (groups IZ-FT and AL-FT) showed total fracture. In these cases, the flaw originated in the framework material surface and propagated directly throughout the specimen as if it was a monolithic material (Figure 2). Lateral cracks were observed in the porcelain layer subjected to compression. Specimens from the YZ-FT group showed 70% of partial failure, in which the porcelain under compression failed before fracture of the framework material under tension, resulting in delamination of the porcelain layer. As failure started in the porcelain under compression, the fracture stress could not be calculated using the equation proposed for pure bending (Eq. 5). Therefore, it was not possible to estimate flexural strength values of these specimens. Yet, three specimens from YZ-FT group showed total failure and could fit in the flexural strength formula, resulting in the following strength values: 732 MPa, 1010 MPa and 972 MPa. Nevertheless, these σ_f values were not considered for statistical analysis because of the low n.

Tri-layered specimens also exhibited both failure modes with 80% of the specimens from YZ-TRI group and 50% of the specimens from IZ-TRI group showing similar failure behavior described in Figure 1 for the bi-layered specimens. All specimens from AL-TRI group fractured as a monolithic material, and the critical flaw was located at the surface of the porcelain under tension. There was no planar difference between the fracture surface of the framework and veneer layers adjacent to the interface, suggesting that the flaw propagated catastrophically throughout the structure. Figure 3 shows the fracture surface of a specimen from group IZ-TRI that had total failure.

Compared to VM9 group, the mean residual stress values of YZ-PT and YZ-TRI groups were 4 MPa and -6 MPa, respectively.

Discussion

The material under tension during testing influenced the flexural strength and the fracture mode of the experimental groups. Therefore, the study hypothesis was accepted. For bi-layer design, the specimens tested with the framework subjected to tension showed higher flexural strength than the specimens tested with the porcelain in tension. These findings are in agreement with the results reported in the literature, using mechanical tests and finite element analysis (FEA) [7, 14, 16, 18].

For IZ and AL framework materials, the flexural strength values observed for the monolithic specimens were similar to the values obtained for bi-layered specimens tested with the framework under tension. It has been reported that the maximum tensile stress during testing for both configurations is located in the specimen surface subjected to tension [17]. These observations are in agreement with studies suggesting that areas subjected to high tensile stress concentration in FPDs, such as the connector cervical area, should not be veneered

with porcelain to improve the restoration mechanical performance [14, 16]. Nevertheless, the literature shows some concern about the effect of direct exposure of the framework ceramic material to the oral environment, especially YZ that is susceptible to a phenomenon called low temperature degradation [2, 28, 29].

The failure mode was also similar for monolithic specimens and bi-layered specimens tested with the framework under tension. The flaw origin was located in the framework surface and propagated catastrophically throughout the structure as if it was a monolithic material, except for 70% of the specimens from YZ-FT group that presented partial failure. In these specimens, the porcelain layer under compression failed before the fracture of the framework layer. During loading lateral cracks were produced in the porcelain layer close to the applied load. As the load increased, the porcelain delaminated from the framework material, and the contact area was crushed. Although lateral cracks were also observed for specimens in the IZ-FT and AL-FT groups, the framework layer failed before the large extension or unstable propagation of such cracks. Thus, no delamination was observed in these specimens. Similar behavior was described by Guazzato et al. (2004) [18], in which 80% of the specimens presented failure of the porcelain layer under compression before failure of the YZ material. The porcelain delamination observed for YZ-FT specimens is probably related to YZ superior mechanical properties. YZ has fracture toughness (K_{Ic}) around $6.5 \text{ MPa}\cdot\text{m}^{1/2}$ [30] and flexural strength (σ_f) of 861 MPa. Compared to the results obtained for VM9 ($K_{Ic} = 0.7 \text{ MPa}\cdot\text{m}^{1/2}$ [30] and $\sigma_f = 66 \text{ MPa}$), YZ values are approximately 10 times higher. Therefore, even though the porcelain layer is not located in the area of maximum tensile stress concentration, complex stresses produced during loading may lead to its failure before YZ material.

Liu et al. (2010) [31] evaluated the mechanical behavior of ceramic bi-layered specimens using three-point flexural test and FEA. The authors also observed porcelain delamination for Y-TZP bi-layered specimens. It was concluded, using FEA, that the interfacial delamination was initiated by shear stresses building up along the interface due to the mechanical properties mismatch of the two ceramic components, especially the mismatch of flexural strength. The FEA simulation suggested that it is possible to avoid delamination by increasing the flexural strength of the veneer porcelain to above 300 MPa. They reported that the fracture in both alumina and Y-TZP bi-layered composites were initiated by tension stresses, but the crack propagated in two different ways. For alumina specimens, the failure went straight over the interface, whereas for Y-TZP specimens an additional shear stress concentration was occurring at the interface, which made porcelain to start delaminating from Y-TZP before total failure. These findings are in agreement with the failure mode observed in the present study for YZ-FT and AL-FT groups.

The interface is a critical area for layered structure. The literature reports a poor chemical bonding between Y-TZP and silicate based veneer porcelain that provides easy paths for crack propagation along the interface [32]. In the present study, a bonding agent (Effect Bonder, Vita) was applied on the surface of YZ specimens. According to the manufacturer (Vita), the Effect Bonder is recommended for non-colored Y-TZP substructures to ensure reliable shade reproduction and to improve the bond with Vita VM9 porcelain. However, the material composition is not provided by the manufacturer and the bonding mechanism of this agent is not well known. Fischer et al. [33] evaluated the shear bond strength between veneering ceramics and zirconia-based framework materials and observed that the application of a liner on Y-TZP had no significant effect. In addition, to investigate the role of the bonding agent on the Y-TZP structures, the interface between the framework materials and the porcelain was carefully observed during the fractographic analysis of bi- and tri-layered specimens. It was not possible to identify differences in the microstructural features of the interface between the three tested framework materials. Thus, the authors

believe that the bonding agent had no significant effect on the failure mode of YZ specimens. This may be the reason for Vita removing the Effect Bonder from the market, indicating the use of a coloring liquid or a wash layer that, probably, serve to improve the color appearance and the wettability and bonding to zirconia based ceramics.

Studies also observed that the framework material, when subjected to tension during testing, had significant influence in the flexural strength and failure mode of layered structures [7, 19]. Della Bona et al. (2003) [7] reported similar flexural strength results for monolithic specimens and for bi- and tri-layered specimens tested with the framework material under tension. In that study, the reliability of the layered structures was directly related to the reliability of the framework material. An investigation with bi-layered specimens tested with the framework material under tension found different results when Procera All-Ceram and In-Ceram Alumina systems were evaluated [19]. For Procera AllCeram, monolithic and bi-layered specimens showed similar flexural strength. On the other side, for In-Ceram Alumina, monolithic specimens showed superior strength compared to bi-layered specimens. However, the results obtained for the layered specimens were still higher than the values obtained for monolithic specimens of porcelain, which indicated that In-Ceram Alumina framework material still had a positive influence in the mechanical performance of these structures. The authors attributed the differences found between these ceramic structures to differences in the interface properties related to the composition of the porcelains and the compatibility between the materials [19].

In the present investigation, the difference between the thickness of the framework material for monolithic (2 mm) and bi-layered (FT) structures (1 mm) had no influence in the flexural strength data. This behavior is related to the fact that the area under tension in the three-point flexural test is limited to the lower part of the specimen, located between the two supporting rollers and below the loading roller [2, 34]. Thus, in both configurations, only the framework material was subjected to the maximum tensile stress. These findings were supported by fractographic analysis, since the critical flaw was located in the surface of the framework material for the specimens in these groups. Although one of the purposes of testing bi-layered structures with the framework in tension was to simulate the failure mode of all-ceramic crowns, the stress distribution induced by three-point flexural test is closer to the stress pattern of a FPD connector. The general configuration of a FPD is similar to the three-point flexural test, in which a ceramic bar is only supported in two points and is under flexion when a load is applied. Yet, a crown is a complex system that is completely supported by a multilayered structure (i.e. luting agent, dentin, resin composite), which has a direct effect on the stress distribution [35].

Except for the specimens from YZ-TRI group, there were no differences among the mean flexural strength values obtained for porcelain monolithic specimens, bi-layered specimens with the porcelain under tension and tri-layered specimens. These results suggest that the mechanical behavior of these structures was controlled by the porcelain layer. Failure of the porcelain layer (partial failure mode) was predominant for bi-layered specimens tested with the porcelain in tension, but the total fracture mode was also showed by some tri-layered specimens. Therefore, the framework material, with superior mechanical properties, failed to improve the flexural strength values and to prevent the fracture of the porcelain under tension. The objective of testing bi-layered structures with the porcelain loaded in tension was the same as testing tri-layered structures, to simulate the stress situation to which the pontic of a FPD is subjected in the mouth. Although tri-layered specimens may be more appropriate to simulate the pontic, most studies with layered ceramic structures tested bi-layered specimens with the porcelain in tension.

For bi-layered specimens with the porcelain subjected to tension during testing the critical flaw was located in the porcelain surface. The flaw propagated through the porcelain layer and deflected near the interface, resulting in partial failure of the specimens and in delamination of the porcelain. This behavior was attributed to differences in the elastic modulus and fracture toughness between the framework and veneering materials. In these cases, the stress necessary to produce fracture of the framework material exceeded the interface fracture energy. In addition, FEA of ceramic bi-layered specimens tested in flexure have shown that tensile stresses are also concentrated at the interface if the porcelain is under tension [17, 18]. Another factor to be considered is the presence of residual stresses that could be induced during processing (i.e., porcelain sintering and cooling) [17]. Notwithstanding, it is important to notice that the test was interrupted after failure of the porcelain layer, without fracture of the framework material. Therefore, the testing methodology is also responsible for the similarity among the mean strength values obtained for these layered structures and the porcelain monolithic specimens.

Studart et al. (2007) [3] also observed crack arresting at the veneer-framework interface when a material with high fracture toughness, such as YZ and IZ, was used to produce layered structures. Thus, the fracture of the porcelain layer does not lead to complete fracture of the ceramic restoration but can expose the framework to the oral environment humidity and eventually enhance the susceptibility to subcritical crack growth [3].

For YZ-TRI and IZ-TRI groups, 80% and 50% of the specimens, respectively, showed partial failure, which was similar to the failure mode observed for bi-layered specimens tested with the porcelain under tension (PT). However, all specimens of AL-TRI group fractured as a monolithic material (catastrophic or total fracture). Although the porcelain was subjected to tension in both bi and tri-layered structures, the framework to veneer thickness ratio was different for these configurations. For bi-layered specimens the ratio was 1:1 (veneer:framework), and for tri-layered specimens the ratio was 0.5:1:0.5 (veneer:framework:veneer). Thus, for bi-layered specimens, considering the stress distribution induced by the three-point flexural test, only the porcelain layer was subjected to the maximum tensile stress. Otherwise, for tri-layered specimens, the porcelain thickness was reduced, placing the framework layer closer to the area of tensile stress concentration. Therefore, an increase in the flexural strength values was expected for these specimens. However, only YZ-TRI group showed significantly higher flexural strength.

As the calculated mean residual stress values were relatively low (4 MPa for YZ-PT and -6 MPa for YZ-TRI), the superior mean flexural strength value of YZ-TRI, compared to YZ-PT, was probably not influenced by thermally induced stresses. As mentioned above, for tri-layered specimens, the framework material is also in the area of tensile stress concentration. Different from AL and IZ framework materials, YZ successfully increased the flexural strength of tri-layered specimens.

As mismatches in the CTE (coefficient of thermal expansion) between the materials could also induce residual stresses, the difference between the CTE of each framework-porcelain combination was examined. According to the manufacturer, the CTE values ($10^{-6} \cdot K^{-1}$) for YZ, AL, IZ, VM7 and VM9 were 10.5, 7.3, 7.7, 7.1 and 9.0, respectively, resulting in a mismatch ranging from 0.2 to 1.5 ($10^{-6} \cdot K^{-1}$). A close matching of the CTE, as observed for these materials, is highly desirable. The influence of the thermal mismatch and other physico-chemical interactions on the core-veneer compatibility was previously reported [12].

The main purpose of evaluating tri-layered structures as described in the present study was to reproduce the configuration of a FPD connector, in which the framework material is completely veneered with porcelain. The results showed that the framework material failed

to improve the flexural strength, since the mechanical behavior of these specimens was mainly controlled by the porcelain material. However, these findings should be extrapolated with caution to the clinical situation, since the framework to veneer thickness ratio of a connector is different from the ratio found in the bar-shaped specimens in this study. Clinically, a larger framework (3–4 mm high $\times$ 3–4 mm thick) is veneered with a porcelain layer (0.6 mm). Therefore, partial failure and delamination of the porcelain are more likely to occur before total fracture of the connector.

Conclusion

The material under tension during testing influenced the flexural strength and the failure mode of the experimental groups, confirming the study hypothesis. For IZ and AL framework materials, the mean flexural strength values observed for the monolithic specimens were similar to the values obtained for the corresponding bi-layered FT (framework in tension) specimens. There was no significant difference among the mean flexural strength values of porcelain monolithic specimens, bi-layered PT (porcelain in tension) specimens and tri-layered specimens, except for the specimens from the YZ-TRI group. Failure of the porcelain layer was predominant for bi-layered PT specimens.

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Figure 1. Representative stereomicroscope images of the fracture surface of a specimen from AL-PT group. A – Lateral view of the specimen showing partial failure of the porcelain layer. B – Close up view of image A (white box); the flaw deflected at the framework-porcelain interface.

Figure 2. Representative SEM images of the fracture surface of a specimen from IZ-FT group. A- White arrow indicates the crack origin. B- Close up view of image A (white box); white arrows indicate lateral cracks in the porcelain stressed under compression.

Figure 3.

Representative SEM images of the fracture surface of a specimen from IZ-TRI group. The fracture origin (white arrow) is located in the porcelain layer subjected to tension. B- Close-up view of image A (white box). Wake hackles (in the white circles) show the direction of crack propagation from crack origin.

Table 1

Materials identification

Legend	Material*	Composition	Indication
YZ	Vita In-Ceram YZ	yttria partially stabilized tetragonal zirconia polycrystal	framework
IZ	Vita In-Ceram Zirconia	alumina-based zirconia-reinforced glass infiltrated ceramic	framework
AL	Vita In-Ceram AL	alumina polycrystal	framework
VM7	Vita VM7	feldspathic porcelain	veneer
VM9	Vita VM9	feldspathic porcelain	veneer

* The materials were manufactured by Vita Zahnfabrik, Bad Sackingen, Germany.

Table 2

Specimens classification according to the number of layers (one, two, three) and testing configuration (porcelain or framework in tension).

Material	One layer	Two layers		Three layers
		Porcelain in tension	Framework in tension	
YZ	YZ	YZ-PT	YZ-FT	YZ-TRI
IZ	IZ	IZ-PT	IZ-FT	IZ-TRI
AL	AL	AL-PT	AL-FT	AL-TRI
VM7	VM7			
VM9	VM9			

Table 3

Density (ρ), Young's modulus (E) and Poisson's ratio (ν) of the materials studied, followed by statistical groupings.

	Materials				
	YZ	IZ	AL	VM7	VM9
ρ (g/cm ³)	6.06 (0.01) ^a	4.30 (0.05) ^b	3.95 (0.01) ^c	2.34 (0.08) ^e	2.44 (0.03) ^d
E (GPa)	209.3 (2.1) ^c	246.7 (2.4) ^b	390.4 (8.3) ^a	66.7 (5.5) ^d	66.5 (0.7) ^d
ν	0.32 (0.01) ^a	0.26 (0.01) ^b	0.24 (0.01) ^{bc}	0.23 (0.02) ^e	0.21 (0.01) ^d

* Values followed by the same letter in the line are statistically similar ($p \geq 0.05$).

Table 4

Three point flexural strength (σ_f) values for the experimental groups (MPa). Results presented as median (standard deviation), and statistical grouping (n=10).

Materials	One Layer	Two Layers		Three Layers TRI
		PT	FT	
YZ	861 (92) ^a	57.6 (7.6) ^e	-----**	83.0 (9.9) ^d
IZ	401 (33) ^c	61.2 (9.1) ^e	412 (22) ^{bc}	63.5 (11.8) ^e
AL	474 (59) ^b	65.8 (15.8) ^e	414 (88) ^{bc}	63.0 (7.3) ^e
VM7	69.8 ± (12.0) ^e			
VM9	65.5 ± (8.9) ^e			

* Values followed by the same letter are statistically similar ($p \geq 0.05$).

** Quantitative data from YZ-FT group was not included in the statistical analysis.