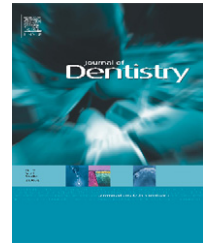


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# Thermo and mechanical cycling and veneering method do not influence Y-TZP core/veneer interface bond strength

Hugo Alberto Vidotti<sup>a,\*</sup>, Jefferson Ricardo Pereira<sup>b</sup>, Elizeu Insaurralde<sup>c</sup>, Ana Lúcia Pompéia Fraga de Almeida<sup>a</sup>, Accácio Lins do Valle<sup>a</sup>

<sup>a</sup>Department of Prosthodontics, Bauru School of Dentistry, University of São Paulo, Bauru, SP, Brazil

<sup>b</sup>School of Dentistry, University of Southern Santa Catarina, Tubarão, SC, Brazil

<sup>c</sup>Department of Prosthodontics and Restorative Dentistry, School of Dentistry, University of South Mato Grosso, Campo Grande, MS, Brazil

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## ABSTRACT

**Objectives:** The purpose of this study was to evaluate the influence of thermal and mechanical cycling and veneering technique on the shear bond strength of Y-TZP (yttrium oxide partially stabilized tetragonal zirconia polycrystal) core–veneer interfaces.

**Materials and methods:** Cylindrical Y-TZP specimens were veneered either by layering ( $n = 20$ ) or by pressing technique ( $n = 20$ ). A metal ceramic group (CoCr) was used as control ( $n = 20$ ). Ten specimens for each group were thermal and mechanical cycled and then all samples were subjected to shear bond strength in a universal testing machine with a 0.5 mm/min crosshead speed. Mean shear bond strength (MPa) was analysed with a 2-way analysis of variance and Tukey's test ( $p < 0.05$ ). Failure mode was determined using stereomicroscopy and scanning electron microscopy (SEM).

**Results:** Thermal and mechanical cycling had no influence on the shear bond strength for all groups. The CoCr group presented the highest bond strength value ( $p < 0.05$ ) ( $34.72 \pm 7.05$  MPa). There was no significant difference between Y-TZP veneered by layering ( $22.46 \pm 2.08$  MPa) or pressing ( $23.58 \pm 2.1$  MPa) technique. Failure modes were predominantly adhesive for CoCr group, and cohesive within veneer for Y-TZP groups.

**Conclusions:** Thermal and mechanical cycling, as well as the veneering technique does not affect Y-TZP core–veneer bond strength.

**Clinical significance:** Different methods of veneering Y-TZP restorations would not influence the clinical performance of the core/veneer interfaces.

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## 1. Introduction

Metal ceramic restorations for fixed prosthodontics treatment have been widely used and proved as a reliable option since the early 1960s. However, the growing demand for aesthetic treatment in recent years has lead clinicians to look for more aesthetic materials. As a result, various all-ceramic materials

and manufacturing techniques have been developed, since they have great aesthetic potential due to their improved optical properties such as translucence, transmission and diffusion of light.<sup>1,2</sup>

All-ceramics often have the same design of metal ceramic restorations: a bilayered structure consisting of a core material with improved mechanical properties veneered by a brittle glass ceramic with improved aesthetic appearance. Yttrium

\* Corresponding author at: Department of Prosthodontics, Al. Octávio Pinheiro Brisola, 9-75, Bauru, SP, ZIP Code 17012-901, Brazil. Tel.: +55 14 81223919.

E-mail address: [hvidotti@yahoo.com.br](mailto:hvidotti@yahoo.com.br) (H.A. Vidotti).

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oxide partially stabilized tetragonal zirconia polycrystal (Y-TZP) systems are widely used as core material since it shows superior mechanical properties to other ceramics due to a transformation toughening mechanism.<sup>3,4</sup> *In vitro* studies showed that Y-TZP has high flexural strength (900–1200 MPa) and fracture toughness (9 to 10 MPa m<sup>1/2</sup>).<sup>5–7</sup> Since Y-TZP does not have a glassy phase due to its polycrystalline structure, veneering with a glass ceramic is imperative to achieve aesthetics.<sup>8</sup>

Clinical performance of all-ceramics bilayered restorations rely on factors such as modulus of elasticity and different coefficient of thermal expansion (CTE) between materials, the core design to provide adequate support for the veneering glass ceramic, and the bonding between core and veneer material.<sup>9,10</sup> The latter is a key factor for success and is related to clinical failure like veneering porcelain delamination that may lead to catastrophic failure and need for restoration replacement.<sup>11,12</sup>

Indeed, chipping of the veneering porcelain is considered a major concern for Y-TZP prostheses, since its occurrence is often reported in clinical trials.<sup>13–15</sup> The extent of the fracture is related with the survival and technical complications of the restorations. Minor chipping consists of fractures that can be polished or repaired with a resin-based composite. A restoration showing chipping that consists of larger fractures involving functional areas that would lead to a significant alteration of the original anatomy after adjustments are considered failure, since the only treatment option is replacement of the prostheses, which represents a costly outcome for both patients and dental practitioners.<sup>16</sup>

Advances in ceramics processing brought up a new technique to manufacture bilayered zirconia indirect restorations. Instead of traditional handily build-up with porcelain powder mixed with liquid (layering technique), the porcelain can be applied over the core with a pressing procedure based on the lost wax technique that would facilitate the veneering method, minimising the presence of internal defects like air bubbles and improving the wetting between veneer and core material.<sup>17</sup> However, few studies were conducted to compare the bond strength between different veneering techniques on zirconia core/veneer interface.<sup>18,19</sup>

In the oral environment, restorations are subjected to moisture, mechanical and thermal fatigue that induce temporary deformations and internal stresses within the materials and their interfaces. Aqueous environment is essential to simulate mechanical cycling in ceramic materials,

since the presence of water can act chemically at crack tips and decrease the strength of ceramics, thus having influence on static strength and cyclic loading tests.<sup>20</sup> In spite of that, simulated thermal and mechanical cycling is rarely performed on bond strength experiments.<sup>21</sup> Until now, no study was conducted to determine the performance of Y-TZP core/veneer interface by thermal and mechanical induced stresses.

So, the purpose of this study was to investigate the influence of the application method (layered *versus* pressed veneer) and of thermal and mechanical cycling on the shear bond strength of Y-TZP core/veneer interface. The null hypothesis tested was that thermal and mechanical cycling and application method would not influence the core/veneer bond strength.

## 2. Materials and methods

### 2.1. Specimen preparation

All-ceramic substructure material tested was Y-TZP machined by the CAD/CAM technique (IPS e.max ZirCAD<sup>®</sup>, Ivoclar-Vivadent, Schaan, Liechtenstein). A glass-ceramic veneer (IPS e.max Ceram, Ivoclar-Vivadent, Schaan, Liechtenstein) was used for layering technique ( $n = 20$ ) and a pressable glass ceramic veneer (IPS e.max Zirpress, Ivoclar-Vivadent, Schaan, Liechtenstein) was used for pressing technique ( $n = 20$ ). An additional metal ceramic system (CoCr) (Fit Cast CoCr<sup>®</sup>, Talladium, Valencia, EUA) was used as control ( $n = 20$ ) and layered with a compatible glass ceramic veneer (IPS Inline<sup>®</sup>, Ivoclar-Vivadent, Schaan, Liechtenstein). Table 1 shows the materials used and their chemical composition.

A cylindrical stainless steel matrix was used for specimen preparation, ceramic layering and shear strength testing. The matrix had a central hole with 6.5 mm in depth and 6.0 mm in diameter. A disc (6.0 mm in diameter; 2-mm thick) was used as a spacer to standardize the veneer layer thickness.

The wax patterns were made with the disc positioned inside the perforation. The wax was liquefied at 75 °C and flowed using a dropper into the perforation. After wax cooling, the patterns were removed using a metallic pin in an auxiliary perforation and stored in water until the investment/casting procedures for metal-ceramic group. For the Y-TZP groups a wax pattern was scanned and patterns were milled in a CAD/CAM system (Cerec InLab<sup>®</sup>, Sirona, Bensheim, Germany).

**Table 1 – Different materials used in this study, their chemical composition and physical properties.<sup>a</sup>**

Material	Chemical composition (% mass)	Flexural resistance (MPa)	Coefficient of thermal expansion (10 <sup>-6</sup> K <sup>-1</sup> )
IPS e.max ZirCAD	ZrO <sub>2</sub> = 87; Y <sub>2</sub> O <sub>3</sub> = 4; HfO <sub>2</sub> = 1; Al <sub>2</sub> O <sub>3</sub> = 0.5	900	10.75
Fit Cast CoCr	Co = 61; Cr = 30; Mo = 5.9; Si < 1; Mn < 1	721	14
IPS e.max Ceram	SiO <sub>2</sub> = 60; Al <sub>2</sub> O <sub>3</sub> = 8; Na <sub>2</sub> O = 6 K <sub>2</sub> O = 6; ZnO = 2; CaO = 2; P <sub>2</sub> O <sub>5</sub> = 1; F = 0.5	90	9.5
IPS e.max ZirPress	SiO <sub>2</sub> = 57; Al <sub>2</sub> O <sub>3</sub> = 12; Na <sub>2</sub> O = 7 K <sub>2</sub> O = 6; CaO = 2; ZrO <sub>2</sub> = 1.5; P <sub>2</sub> O <sub>5</sub> = 1; F = 0.5	110	9.75
IPS Inline	SiO <sub>2</sub> = 59.5; Al <sub>2</sub> O <sub>3</sub> = 13; K <sub>2</sub> O = 10; Na <sub>2</sub> O = 4	80	12.6

<sup>a</sup> According to manufacturers information.

**Table 2 – Firing procedures of the dental ceramics tested.**

Cerâmicas	iT (°C)	dT (min)	t↑ (°C/min)	fT (°C)	hT (min)	oT (°C)
Fit Cast CoCr + IPS Inline (CoCr)						
Opaque	403	6	100	930	2	–
Dentine	403	4	60	910	4	–
Glazing	403	6	60	850	1	–
IPS e.max ZirCad + IPS e.max ZirPress (Y-TZP Pressing)						
Zirliner	403	4	40	960	1	–
Pressing	700	–	60	910	15	–
Glazing	403	6	60	725	1	450
IPS e.max Zircad + IPS e.max Ceram (Y-TZP Layering)						
Zirliner	403	4	40	960	1	–
Wash	403	4	40	750	1	–
Dentine	403	4	40	750	1	–
Glazing	403	6	60	725	1	450

iT, initial temperature; dT, drying time; t↑, temperature raising rate; fT, final temperature; hT, holding time; oT, furnace open temperature.

Veneering was carried out with layering or pressing technique following the manufacturer’s instructions for mass preparation, condensing, baking temperature and time (Table 2).

**2.2. Thermal and mechanical cycling**

Ten specimens from each group were subjected to both thermal and mechanical cycling. The specimens were first thermocycled for 6000 cycles between 5 and 55 °C in deionized water (Elquip, São Carlos, Brazil) with dwell time of 15 s and transfer time of 5 s. After that, specimens were mechanically cycled in aqueous environment at 37 °C with a mechanical cycling machine (Elquip, São Carlos, Brazil) with a 3.2 mm diameter indenter inducing 50 N load for 20,000 times with a frequency of 1 cycle per second. The loading was applied axially on the centre of the porcelain portion of the specimen. The remaining 10 specimens were stored in distilled water for 24 h at 37 °C prior to shear bond strength test.

**2.3. Shear bond strength test**

The specimens were positioned into the matrix with the disc at the bottom of the perforation, leaving the ceramic layer visible outside the matrix, such that the shear forces could only be applied at the interface. Shear strength testing was performed in a universal testing machine (Emic, São Paulo, Brazil) with a 0.5-mm thick bevel-shaped rod at a crosshead speed of 0.5 mm/min until failure. Data were analyzed by two-way analysis of variance (ANOVA) at 5% significance level. Multiple comparisons were made by Tukey’s adjustment test.

**2.4. Fracture surface analysis**

Fracture analysis was performed on stereomicroscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany) and scanning electron microscope (JSM, 220A, JEOL, Tokyo, Japan).

Failure modes were classified as<sup>22</sup>:

CV. Cohesive failure in the veneer.

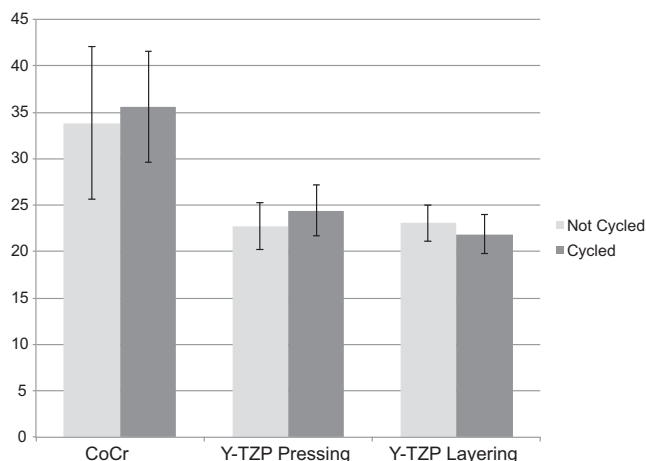
M. Mixed fracture starting within the veneer, crossing it and continuing into the interface.

A. Adhesive failure at the core/veneer interface.  
CC. Cohesive failure within the core.

**3. Results**

The results showed no statistical differences in shear bond strength values before and after thermo and mechanical cycling for all groups (Fig. 1). None of the specimens tested showed debonding during ageing procedures. Significant shear bond strength difference was found between the materials tested ( $p < 0.05$ ). (Table 3). Tukey’s multiple comparisons test and mean shear bond strength results for each group are shown in Table 4.

Fracture analysis is presented in Table 5 and distinct failure modes were found for each group. The metal–ceramic group (control) exhibited predominately adhesive failure. SEM analysis showed that there was a thin layer of glass ceramic attached on YTZ-P core of the specimens in both groups (Figs. 2 and 3) and failure mode was predominantly cohesive within the veneering porcelain (Table 5). Only one specimen from group YTZ-P Pressing showed cohesive fracture within the core.



**Fig. 1 – Shear bond strength results with and without ageing for the materials combinations tested (MPa).**

**Table 3 – Results of two-way analysis of variance for the cycling fatigue conditions, materials, and interaction according to shear bond strength data ( $p < 0.05$ ).**

Effect	DF	MS	F	p
Materials	2	918.77	43.89	<0.01
Cycling	1	7.64	0.36	0.548
Interaction	2	14.40	0.68	0.507

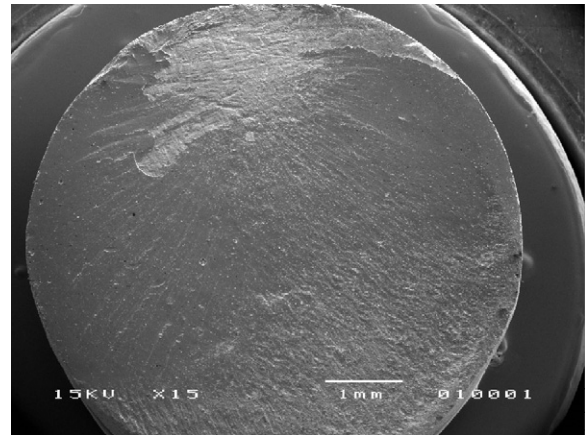
#### 4. Discussion

The results of this study accepted the null hypotheses that the application method would not influence the core/veneer bond strength and that those interfaces would not be affected by thermal and mechanical cycling.

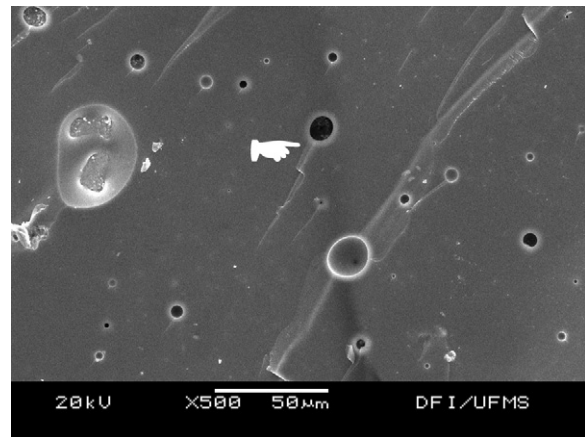
The thermal and mechanical cycling procedures were based on previous studies available in the literature.<sup>23,24</sup> Vasques et al.<sup>23</sup> using a very similar protocol, found a reduction in mean shear bond strength of TiCP core/porcelain veneer interface, but not for noble alloys. On the other hand, other studies on different all-ceramic materials showed that thermocycling alone did not influence the bond strength of core/veneer interface.<sup>8,25,26</sup> Thermo and mechanical cycling combined have never been performed on Y-TZ-P. Since no reduction in bond strength was found in the present study, it can be suggested that those interfaces are stable in the presence of mechanical and thermal stresses in moisture, like in the oral environment.

Significant difference was found between the shear bond strength of the materials, and the control group (metal-ceramic) showed significantly higher shear bond strength values ( $p < 0.01$ ). The mean values were similar to the findings of Al-Dohan et al.<sup>1</sup> and Guess et al.<sup>8</sup> These authors also found statistically differences between zirconia and metal ceramic cores and veneering porcelain interface. Although the different veneering materials used for the control and the Y-TZP groups have similar chemical composition and mechanical properties, internal stresses induced by the Y-TZP core material in the veneering porcelain may have influenced the cohesive resistance of the latter. The role of thermal behaviour between bonded materials in bilayered all ceramic restorations is well known.<sup>27</sup> Coefficient of thermal expansion (CTE) mismatch of the materials leads to decrease in bond strength or to spontaneously debonding during firing, especially when the CTE of the veneering porcelain is higher than the core material.<sup>28,29</sup>

In spite of the control group (metal-ceramic) demonstrating significantly higher shear bond strength values, careful examination of the failure mode revealed that both



**Fig. 2 – SEM image of Y-TZP specimen showing a cohesive fracture of the veneering porcelain.**



**Fig. 3 – Greater magnification of fractured Y-TZP specimen showing a thin layer of veneering porcelain attached to the core. The presence of wake hackles (pointer) indicates the direction of the propagation of the fracture.**

experimental groups showed almost exclusive cohesive failure within the veneering porcelain. Fractography analysis showed that the fracture started near the region where the force was applied and propagated in the opposite direction along the porcelain, near the interface, as evidenced by the wake hackles founded (Fig. 3). That suggests that it was not the bonding *per se* that had influence on the lower values of shear bond strength, but inherent factors within the veneering porcelain.

**Table 4 – Mean shear bond strength (MPa) of all-ceramic specimens and control group (CoCr).**

Grupo	Materials		n	Mean	SD	*
	Core	Veneer				
CoCr	Fit Cast CoCr	IPS Inline	20	34.7	±7.1	a
Y-TZP Pressing	IPS e.max ZirCAD	IPS e.max ZirPress	20	23.6	±2.1	b
Y-TZP Layering	IPS e.max ZirCAD	IPS e.max Ceram	20	22.5	±2.1	b

\* Means followed by the same letter in a column are not significantly different according to Tukey's test at 5% probability level.

**Table 5 – Failure modes for each group (values in %): (CV) cohesive failure in the veneer, (M) mixed fracture, (A) adhesive failure at the core/veneer interface, (CC) cohesive failure in the core.**

Group	Failure mode			
	A	CV	CC	M
CoCr	85	5	–	10
Y-TZP Pressing	–	95	5	–
Y-TZP Layering	–	100	–	–

In the present study the veneering porcelain bonded to the core materials showed compatible CTE. Thermal conductivity also influences the mechanical behaviour of veneering porcelain. Zirconia has a low thermal conductivity behaviour and leads to residual stress accumulation on the adjacent veneering porcelain during cooling after firing.<sup>27</sup> Komine et al.<sup>30</sup> showed an increase in shear bond strength between zirconia and veneering porcelain when a slow cooling protocol, instead of automatically opening of the furnace, was performed. In both cases fractures were cohesive within the veneering porcelain with a thin layer of glass still attached to the zirconia core material near the interface, similar to the findings founded in the present study. They attributed that increase in bond strength to lower residual stresses in the veneering porcelain, just near the interface, due to slow cooling protocol that diminished the stress concentration caused by the zirconia conductivity behaviour. So, the lower values of shear bond strength of the Y-TZP groups may be explained by residual stresses presented in the veneering porcelain just near the interface. This may also explain the presence of a thin layer of glass ceramic attached to the Y-TZP cores very close to the interface founded in the fracture analysis with SEM (Fig. 3).

The veneering method for Y-TZP did not influence the nominal shear bond strength values. These results are in accordance with Aboushelib et al.<sup>18</sup> but not with Lopez-Molla et al.<sup>19</sup>, who found slightly higher bond strength values for the pressing technique. The authors attributed that to a possible closer contact of the veneering porcelain to the Y-TZP core surface due to the high pressure achieved in the pressing procedure that would lead to a lesser concentration of voids at the interface. However, in that study, failure mode analysis was not performed with either optical or with scanning electron microscopy. In the present study failure mode and the presence of pores and voids on the fractured surfaces were similar for both Y-TZP veneering techniques tested. The present results suggest that the method of veneering have no influence on the bond strength between Y-TZP and veneering glass ceramic.

One limitation of this study was that the authors only tested materials from one manufacturer. Further research must be conducted to investigate the influence of thermal and mechanical cycling on the bond Y-TZP core/veneer bond strength of other manufacturers. Studies on reducing internal stresses within the veneering porcelain for Y-TZP core materials are also suggested.

## 5. Conclusions

The interfaces of veneering glass ceramics to Y-TZP core material tested have not showed decrease in bond strength when subjected to thermal and mechanical stresses in a wet environment. It is also not affected by the veneering method (layering or pressing technique) for the Y-TZP core material tested.

The different materials showed significant difference of shear bond strength. The control group (CoCr) presented the significantly higher results for shear bond strength.

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