

BONDING TO DENSELY SINTERED ALUMINA- AND GLASS INFILTRATED ALUMINUM / ZIRCONIUM-BASED CERAMICS

ADESÃO A UMA CERÂMICA ALUMINIZADA DENSAMENTE SINTERIZADA E A UMA CERÂMICA A BASE DE ALUMINA / ZIRCÔNIA INFILTRADA DE VIDRO

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Received: March 1, 2004 - Modification: April 19, 2004 - Accepted: June 1, 2004

ABSTRACT

The objective of this study was to test two hypotheses: (1) silica coating affects the bond strength between ceramics and a resin cement; (2) bond strength is affected by the type of ceramic. Twelve blocks 5 x 6 x 8 mm of In-Ceram Zirconia (ZR) and twelve Procera AllCeram (PR) ceramics were made and duplicated in composite. Five blocks of each ceramic were treated as follows: (1) ZR + GB (laboratorial airborne particles abrasion with Al₂O₃ particles) + silane; (2) ZR + SC (chairside tribochemical silica coating system, Cojet); (3) PR + GB; (4) PR + SC. Two treated samples of ceramic were analyzed under SEM. The ceramic-composite blocks were cemented with Panavia F and stored in 37°C distilled water for 7 days. They were then cut to produce bar specimens (n=30) with a bonding area of 0.6±0.1mm². Specimens were loaded to failure under tension in a universal testing machine (1 mm/min). Bond strength (σ) values were statistically analyzed using ANOVA (Two-way) and Tukey ($\alpha = 0.05$). Mean σ (MPa) and standard deviation were as follows: 1) 15.1 ± 5.3; 2) 26.8 ± 7.4; 3) 12.7 ± 2.6; 4) 18.5 ± 4.7. Silica coated surfaces showed statistically higher σ than the same substrate treated with GB only. In addition, ZR (with vitreous phase) showed higher σ than PR (without vitreous phase).

Uniterms: Ceramics; Surface treatment; Bond strength; Microtensile test.

RESUMO

Objetivo deste estudo foi testar duas hipóteses: (1) a deposição de sílica afeta a resistência adesiva entre cerâmicas e cimento resinoso; (2) a resistência adesiva é afetada pelo tipo de cerâmica. Dez blocos (5 x 6 x 8 mm³) das cerâmicas In-Ceram Zircônia (ZR) e Procera AllCeram (PR) foram confeccionados e duplicados em resina composta. Cinco blocos de cada cerâmica foram assim tratados: (1) ZR + GB (jateamento com partículas de Al₂O₃) + silano; (2) ZR + SC (deposição de sílica/silanização – Sistema CoJet); (3) PR + GB; (4) PR + SC. Os blocos de cerâmica-compósito foram cimentados com Panavia F e armazenados em água destilada (37°C / 7 dias). Eles foram então cortados para obter corpos-de-prova em forma de barras (n=30) com uma área adesiva de 0,6 ± 0,1mm². Os cp foram submetidos ao teste de microtração em uma máquina de ensaio universal (1mm.min⁻¹). Os valores de resistência adesiva (σ) foram submetidos à análise de variância (2 fatores) e ao teste de Tukey ($\alpha = 0,05$). As médias de σ (MPa) e os desvios padrão foram: 1) 15,1 (5,3); 2) 26,8 (7,4); 3) 12,7 (2,6); 4) 18,5 (4,7). A deposição de sílica na superfície cerâmica apresentou maior que o mesmo substrato tratado com GB. Além disso, ZR (com fase vítrea) apresentou maior σ que PR (sem fase vítrea).

Unitermos: Cerâmica, Tratamento de superfície, Resistência adesiva, Teste de microtração.

INTRODUCTION

The metal-free ceramic systems appeared when the content of aluminum oxide was increased in the feldspar-based ceramic¹⁸, increasing its mechanical properties and allowing the ceramic restorations to be used in the posterior teeth.

In-Ceram Alumina ceramic is composed of approximately 80% aluminum oxide (Al_2O_3) – crystalline phase – infiltrated by 20% of lanthanum oxide – vitreous phase¹⁴. The densely sintered alumina ceramic (Procera AllCeram) presents a high content of Al_2O_3 (99.9%)¹. In-Ceram Zirconia ceramic presents approximately 67% of Al_2O_3 , 13% of ZnO, and 20% of lanthanum oxide (vitreous phase). Although these ceramics present different compositions, they are considered resistant substrates to surface degradation by hydrofluoric acid^{1,21}.

Several studies have reported that the bonding mechanism between feldspar ceramics (silica-based ceramics with low content of Al_2O_3) and resin cements starts by the etching with hydrofluoric acid and is optimized by the silane coupling agent. The etching and silanization increase the surface energy and the wettability of the ceramic substrate and decrease the contact angle between the ceramic surface and the resin cement²⁴. Besides, the silane agents present bi-functional characteristics, i.e., they allow bonding between the silica of the feldspar ceramic and the organic matrix of the resinous materials through siloxane bridges^{7,13,24}.

The tribochemical silica coating in the ceramic surface seems to be one of the best methods to promote the bonding of acid-resistant ceramics to resin cements^{1,2,10,30}. However, no other study has investigated the effect of surface treatments of different acid-resistant ceramics on the bonding to a resin cement.

Thus, the purpose of this study was to test the following hypotheses: (1) tribochemical silica coating affects the bond strength between acid-resistant ceramics and a resin cement; (2) bond strength is affected by the type of ceramic (with vitreous phase and without vitreous phase).

MATERIAL AND METHODS

Twelve ZR ceramic blocks (In-Ceram[®] Zirconia, VITA Zahnfabrik, Bad Säckingen, Germany) (glass-infiltrated alumina/zirconia ceramic) and twelve PR ceramic blocks (Procera AllCeram, Nobel Biocare, Stockholm, Sweden) (high-purity alumina ceramic), all measuring 5 x 6 x 8mm, were fabricated and supplied by the manufactures. All cementation surfaces of ceramic blocks were polished using 600- to 1200-grit metallographic paper # 600, 800 and 1200 (3M, St. Paul, USA) in a polishing machine (Labpol, Extec, USA). Each ceramic block was duplicated in composite resin (W3D Master, Wilcos, Petrópolis, Brazil – batch # 02902) using a mold of impression material (Elite HD, Zhermack, Badia Polesine, Italy; batch # B01.01.B). Inside the mold, composite resin layers were condensed (2mm) and light

cured for 40s (XL 3000 - 3M/ESPE, St. Paul, USA; light intensity = 500mW/cm²) until completion of the mold. One composite resin block (RC) was obtained for each ceramic block.

The ceramic surfaces of the 10 blocks of each ceramic (ZR and PR) were divided into groups (5 per group) according to the surface treatments:

GB: Laboratorial airborne particles abrasion with 110- μ m aluminum oxide particles (Micro-Etcher - Danville Inc., Danville, EUA), considering the following parameters: a) blasting perpendicular to the surface; b) distance: 10mm; c) time: 20s; d) pressure: 2.8 bars^{16,20,25,30}. The silane was applied, waiting 5 min.

SC (CoJet System, 3M/ESPE, Minnesota, EUA; batch # 118484): Chairside airborne particles abrasion with 110- μ m aluminum oxide particles modified with salicylic acid (CoJet-Sand), using the same blasting parameters applied in GB. The surfaces were then coated with a silane coupling agent (ESPE-Sil) and allowed to dry for 5 minutes^{9,11,25}.

Two samples of each ceramics (one for each treatment) were treated and sputter-coated with gold-palladium for 3 min in a Hummer II Sputter Coater (21020, Technics Inc., Alexandria, VA, USA) at a current of 10mA, and vacuum of 130mTorr, and the surface topography was examined using a scanning electron microscope (JSM 6400, Jeol Ltd., Tokyo, Japan).

Each ceramic block was cemented to the composite block using a resin cement (Dual-Cure Dental Adhesive System Panavia F, Kuraray Med Inc., Okayama, Japan, batch # 51.133), which was manipulated according to the manufacturer's specifications, and then applied onto the treated ceramic surface using a Centrix syringe (DFL, Rio de Janeiro, Brazil). The ceramic-cement-composite set was placed on a press with the interface (cementation surface) perpendicular to a vertical load of 750g/10min^{16,20,25,30}. For this time, the excesses were removed and each free block face was cured for 40s using XL 3000 curing light (light intensity = 500mW/cm²; distance = 0mm)⁸. Oxyguard was applied onto the interface margins, and after 10 minutes the blocks were removed from the press, washed with air-water spray, and stored in distilled water at 37°C for 7 days. Four groups containing 5 cemented blocks (Figure 1A) were therefore constituted: (G1) ZR + GB; (G2) ZR + SC; (G3) PR + GB; (G4) PR + SC.

The blocks were bonded with cyanoacrylate glue (Super Bonder gel, Loctite Ltda, São Paulo, Brazil) to an adapted metallic base, which was coupled to a cutting machine (Figure 1B). Slices were obtained using a slow-speed diamond wheel saw (# 7016, KG Sorensen, Barueri, Brazil) under cooling. The first slice of each block - approximately 1mm in thickness - was discarded because the results could be influenced by the excess or absence of resin cement on the interface^{17,26}. Two slices (1 \pm 0.1mm thickness each) were then obtained per block (Figure 1C). Each slice was rotated (90°) and bonded onto the metallic base (Figure 1D). The first bar specimen was also disregarded for the same reasons described above. Three other cuts were made. Non-trimmed rectangular shape samples (bar specimens) with

approximately 0.6 mm² of bonding area. This same procedure was performed in the other slice. Six samples per cemented block were obtained (Figure 1E). Therefore, only the internal samples were used (Figure 1F), so each experimental group was composed of 30 samples^{3,7,20,25}. The adhesive area (“A”) of each sample was measured using a digital caliper (Mitutoyo, Tokyo, Japan) prior to the test.

For the microtensile test, the extremities of each sample were bonded to an adapted caliper using cyanoacrylate glue. The sample was glued parallel to the long axis of the caliper, thus minimizing the bending forces in the adhesive zone. This apparatus was coupled to a universal testing machine (EMIC DL-1000, EMIC, São José dos Pinhais, Brazil), and tensile stress was applied to it (crosshead speed of 1mm.min⁻¹)^{3,7,20,25}.

The bond strength ($\sigma = L/A$) was calculated. “L” is the load in the moment of rupture (Kgf) and “A” is the bonding area of the sample (mm²). The results were submitted to ANOVA (two-way analysis of variance) and Tukey test ($\alpha = 0.05$).

The fractured surfaces of the samples were analyzed in a light microscope – 50x magnification (Zeiss MC 80 DX,

Zeiss, Jena, Germany) – to assess the failure mode (adhesive, cohesive or mixed).

RESULTS

The mean bond strength values and the standard deviation of the experimental groups are presented in Table 1. It was possible to note that: (1) the interaction effect of the two variables: ceramic and surface treatment ($F_{df(1,116)} = 9.08$; $p = 0.003$); (2) the ceramic effect ($F_{df(1,116)} = 30.74$; $p = 0.001$); (3) the surface treatment effect ($F_{df(1,116)} = 81.44$; $p = 0.001$). According to the Tukey test, the bond strength of G2 was statistically higher than the other groups. G1 and G4 were statistically similar.

The mean values of bond strength of the surface

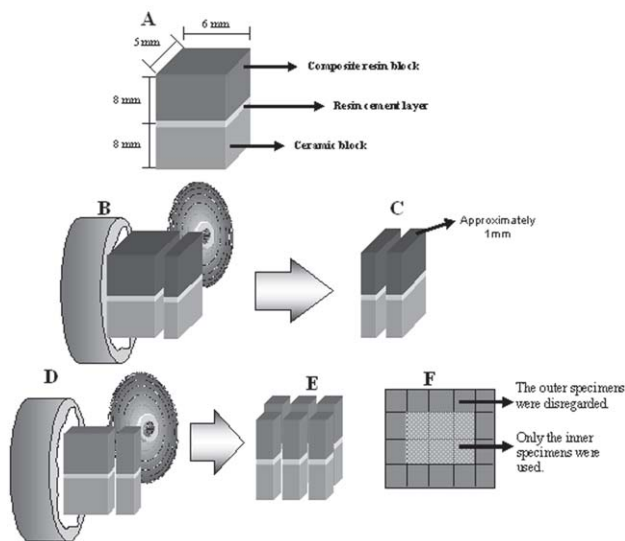


FIGURE 1- (A) Cemented ceramic and polymeric blocks. (B) A slice cut of from the block fixed to the cutting machine. (C) Internal slices to be cut again. (D) Samples being obtained. (E) Internal samples obtained and used in this study (F)

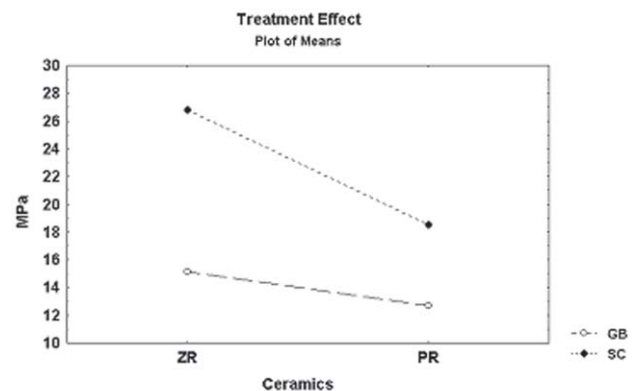


FIGURE 2- Surface treatment effect

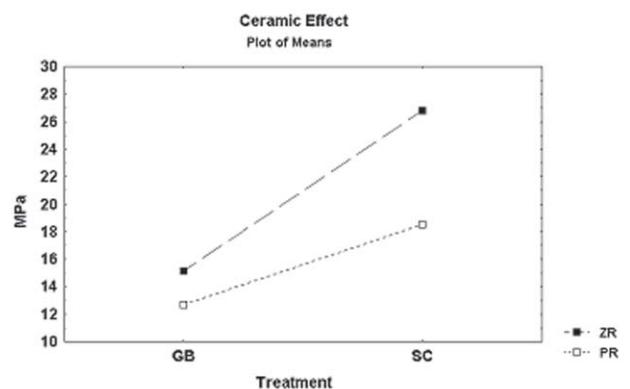


FIGURE 3- Ceramic effect

TABLE 1- Mean values of bond strength and standard deviations (SD) of each group. The interactions of the factors are shown

Surface treatment	Ceramics		Main effect surface treatment
	PR	ZR	
GB	12.7 (2.6) ^a	15.1 (5.3) ^{ab}	13.925 (4.3)
SC	18.5 (4.7) ^b	26.8 (7.4) ^c	22.667 (7.4)
Main effect ceramic	15.6 (4.8)	20.9 (8.7)	

*Different superscript letters mean statistical difference

treatment factor are presented in Table 1 and in Figure 2. When the treatment factor is analyzed in Table 1, one can observe that the CoJet System (tribochemical silica coating) presented statistically higher bond strength than the Al_2O_3 -treatment.

The mean bond strength values of the ceramic factor are described in Table 1 and Figure 3. Analyzing this Table, one can conclude that the In-Ceram Zirconia (ZR) ceramic presented higher bond strength than that of the Procera ceramic, but that is dependent on the surface treatment.

The SEM images of the two ceramics submitted to surface treatments are shown in Figure 4. The topographic analyses of these SEM micrographs suggest that airborne abrasion with the SiO_x particles (Figures 4B and 4D) promoted silica coating (SC) on the surface. The topographic patterns for 4B and 4D are different when compared to blasting with 110- μm aluminum oxide particles (Figures 4A and 4C). The micro-retentive pattern observed after blasting with Al_2O_3 particles (GB) seems to contribute to the micromechanical bond when compared to the topographic pattern observed in the treatment with airborne abrasion with SiO_x particles (silica coating) (SC), albeit the SC have allowed higher bond strength.

All samples tested were analyzed in light microscope (50x magnification). Notwithstanding the groups investigated, all samples (100%) presented adhesive failure mode in the adhesive zone (interface resin cement / ceramic or resin cement).

DISCUSSION

In Table 1, it is possible to observe that G2 (In-Ceram Zirconia ceramic treated with chairside tribochemical silica coating) presented the highest value of bond strength. G1 (ZR + GB) presented bond strength similar to G4 (PR + SC).

These results may be explained by the following phenomena: (1) there is a chemical bond between coated silica, silane agent, and resin cement^{15,21}; (2) there is a chemical bond of the MDP monomers-phosphate of the resin cement to the aluminum- and zirconium-oxides^{29,30}; (3) the presence of a vitreous phase in the In-Ceram Zirconia ceramics facilitates the silica coating, and therefore increases the bond strength.

Considering the ceramic factor (Table 1), notwithstanding the surface treatment, one can observe that the In-Ceram

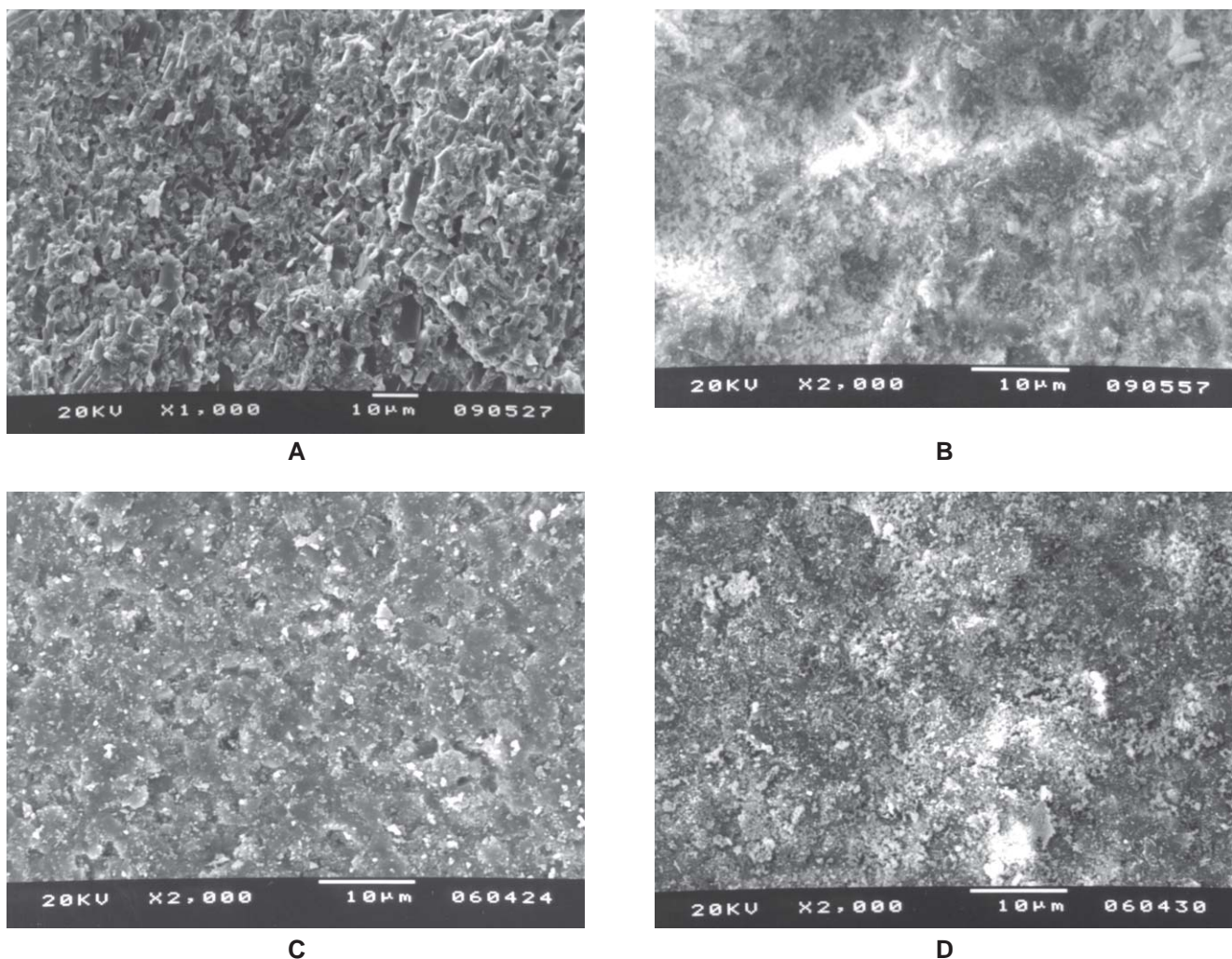


FIGURE 4- Treated ceramic surface of In-Ceram Zirconia (ZR): **A** – Al_2O_3 particles; **B** – SiO_x particles (x2000). Treated ceramic surface of Procera AllCeram (PR): **C** – Al_2O_3 particles; **D** – SiO_x particles (x2000)

Zirconia ceramics presented higher bond strength than the Procera AllCeram ceramic. ZR is composed of 67% aluminum oxide, 13% zirconium oxide, and 20% vitreous phase composed of lanthanum oxide¹⁴, whereas Procera is composed of 99.9% alumina, without vitreous phases¹. Although there are no other comparative studies available, our study corroborates with the trend that there is higher bond strength between resin cement and glass infiltrated aluminous ceramic treated with tribochemical systems^{14,20,21,25}. Thus, one can assume that the presence of a vitreous phase in the In-Ceram Zirconia (ZR) ceramic assists the silica coating on the ceramic surface.

The dense microstructure of the Procera AllCeram ceramic restricts a reliable bonding to the resin cement without monomer-phosphate, when this ceramic is treated with conventional methods (hydrofluoric acid or blasting with Al_2O_3)^{1,2}. However, Friederich and Kern¹⁰ observed better bond strength results when using a resin cement with monomer-phosphate.

According to Wegner, et al.²⁹, the yttrium-oxide-partially-stabilized zirconia ceramic (Y-TZP) contains high crystalline content (94.9% zirconium oxide stabilized by 5.1% yttrium oxide). It was reported that this ceramic presented an adhesive performance similar to the Procera AllCeram ceramic. For instance, when Y-TZP is blasted with Al_2O_3 and is cemented to a resin cement containing monomer-phosphate, a higher and stable bond strength is obtained^{29,30}.

Although some studies have evaluated ceramics with different microstructures, it can be said that the Y-TZP ceramic and the densely sintered alumina ceramic (PR) are compact materials without vitreous content^{10,29,30}, therefore characterized as acid-resistant ceramics and resistant to silica coating. In this study, we were able to observe this tendency because we compared one densely ceramic without vitreous phase (PR) and one ceramic with vitreous phase (ZR).

Regardless the ceramic being studied, our results confirm that the chairside tribochemical silica coating systems presented a statistically higher bond strength when compared to the blasting with Al_2O_3 .

The bonding to the ceramic substrate has typically been based in the relationship between the silica of the ceramic and silane agent²⁴ (acid-sensitive). The silane presents a bi-functional characteristic, i.e., it allows bonding between the silica of the ceramic and the organic matrix of the resin cements through covalent bridges (siloxane bonds). Besides, silane coupling agents increase the surface energy and wettability, improving the microscopic interaction between ceramics and resin cements^{7,13,24}. The bonding capacity of silanes with silica is very well established in the acid-sensitive ceramics (feldspar-, leucita-, and lithium disilicate-based ceramics). The basic chemical reaction between the silane agent and the ceramics is obtained by the reaction between the γ -methacryloxypropyltrimethoxy-silane (γ -MPTS) and the siliceous oxide present in the surface, i.e., the silanes promote a chemical bond via cross-link with methacrylate grouping of the resinous materials¹³. This silica-silane chemical bond can also occur with acid-resistant ceramics, provided that the silica coating of the ceramic

surface is used¹⁶.

The topographic analyses of the treated ceramic surfaces suggest that the airborne abrasion with SiO_x particles (Figures 5B and 5D) promoted silica coating on the surface, allowing chemical bond between coated silica – silane – resin cement. This topographic pattern is different from that obtained by blasting with Al_2O_3 particles alone (Figures 5A and 5C). This is corroborated by the work of Kern and Thompson¹⁶. They observed an increase of the silica content in the surface of the In-Ceram ceramic (15.8% - 19.7%) after treatment with the 110 μ m SiO_x particles (Rocatec-Plus). The samples treated with Al_2O_3 particles (Rocatec-Pre) did not present an increase in the silica content. The authors suggested that the silicated surface by SiO_x particles (Rocatec System) could develop a better bond strength between the In-Ceram ceramic and the resin cement, which was later observed in the studies by Kern and Thompson¹⁴, Özcan, et al.²¹, and Valandro, et al.²⁵.

Hence, it is essential to consider the following: a surface treatment can improve greater bond strength to some all-ceramic systems, but not to all. Although the tribochemical silica coating process may be considered an important mechanism to promote bonding^{14,21,25}, the current study observed that this conditioning method promoted a larger increase in bond strength in the acid-resistant ceramic with vitreous phase (ZR), when compared to densely sintered alumina ceramic without vitreous phase (PR).

The results confirm the hypotheses initially proposed: (1) tribochemical silica coating increased the bond strength and, (2) the glass-infiltrated alumina/zirconium ceramic (ZR) presented higher bond strength than the densely sintered alumina ceramic (PR) without vitreous phase.

The storage and thermocycling always should be considered to evaluate the bond strength between ceramic and resin cement, because these conditions contribute to the hydrolytic degradation of the resin cement / ceramic interface and the degradation of resin cement due to failure between fillers and matrix. These experimental conditions can contribute to bond strength decrease^{14,30}.

As to the test method, the goal of bond strength in *in vitro* tests (tensile and shear) is the load application in the samples, i.e., to produce stress specifically in the interface between the materials being tested. Thus, for the test to reproduce the real bond strength between an adhesive system and a dental-, metallic-, ceramic-, or polymeric-substrate, it is important that the stress is homogeneously distributed in the bonding interface, regardless of the test method employed. Shear tests have thus been criticized due to the development of non-homogeneous stress in the bonding interface – stressing more the substrate than the interface. This phenomenon prevents an accurate interfacial bond strength measurement and limits further improvements in the bonding systems (underestimated and misinterpreted results), for the failure takes place in the substrate and not in the adhesive zone^{4,28}. The failure mode and fractographic analyses^{3,6,7,19} reduce the risk of data misinterpretation.

Although conventional tensile tests also present some limitations, such as the difficulty of sample alignment in the

universal testing machine and the tendency of a heterogeneous stress distribution at the adhesive interface^{17,27}, this at kind test may be employed because it provides information of global bond strength^{3,5,12}. However, the microtensile test allows (1) appropriate alignment of the samples, (2) more homogeneous distribution of stress, and (3) a more sensitive comparison or evaluation of bond strengths that are similar^{22,23}.

ACKNOWLEDGMENTS

This study was partially supported by Fapesp (São Paulo/SP, Brazil) – Process 2001/13978-1. We thank Wilcos of Brazil for material support and the Prof. Dr. Ivan Balducci by the statistical analysis support. This study is based on a dissertation submitted to the School of Dentistry, Sao Paulo State University at Sao Jose dos Campos (Brazil), as part of the requirements for the M.Sc. degree.

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