

# Influence of Surface Treatment on the Shear Bond Strength of Ceramics Fused to Cobalt–Chromium

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#### Keywords

Shear bond strength; metal–ceramic system; surface treatment.

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### Abstract

**Purpose:** To evaluate the influence of surface treatment on the shear bond strength between a Co-Cr alloy and two ceramics.

**Materials and Methods:** Forty-eight metal cylinders were made (thickness: 4 mm, height: 3.7 mm) according ISO TR 11405. The 48 metallic cylinders were divided into four groups (n = 12), according to the veneering ceramic (StarLight Ceram and Duceram Kiss) and surface treatments: air-particle abrasion with  $Al_2O_3$  or tungsten drill (W). Gr1: StarLight +  $Al_2O_3$ ; Gr2: StarLight + W; Gr3: Duceram +  $Al_2O_3$ ; and Gr4: Duceram + W. The specimens were aged using thermal cycling ( $3000 \times$ , 5 to  $55^{\circ}$ C, dwell time: 30 seconds, transfer time: 2 seconds). The shear test was performed with a universal testing machine, using a load cell of 100 kg (speed: 0.5 mm/min) and a specific device. The bond strength data were analyzed using ANOVA and Tukey's test (5%), and the failure modes were analyzed using an optical microscope ( $30 \times$ ).

**Results:** The means and standard deviations of the shear bond strengths were (MPa): G1 (57.97  $\pm$  11.34); G2 (40.62  $\pm$  12.96); G3 (47.09  $\pm$  13.19); and G4 (36.80  $\pm$  8.86). Ceramic (p = 0.03252) and surface treatment (p = 0.0002) significantly affected the mean bond strength values.

**Conclusions:** Air-particle abrasion with  $Al_2O_3$  improved the shear bond strength between metal and ceramics used.

Although the trend in modern dentistry is to use metal-free restorations, when clinically analyzed, metal-ceramic restorations are still the most frequently used for making fixed partial dentures (FPDs) and single crowns, as these restorations present excellent clinical performance, low cost when compared with metal-free restorations, a simple cementation technique (zinc phosphate cement), and in the great majority of restorative treatments, natural reproduction of the lost dentition. Due to the high cost of precious alloys in the 1970s and progress made in ceramic technology, the use of basic metal alloys as infrastructure materials for FPDs increased considerably,<sup>1</sup> particularly nickel–chrome- (Ni-Cr) and cobalt–chrome- (Co-Cr)based alloys; however, because of adverse effects, such as allergies to the material, shown particularly by alloys con-

taining nickel and beryllium, the use of more biocompatible<sup>2,3</sup> Co-Cr-based alloys has been suggested, since they have shown excellent marginal integrity and an absence of adverse reactions.<sup>2</sup> Furthermore, these alloys allow treatments of excellent quality, because they have very satisfactory mechanical properties, such as hardness, elasticity, and tensile strength.<sup>1</sup>

Even though they have a metal infrastructure, these restorations are subject to failures that could occur predominantly at the interface between the metal and the porcelain.<sup>4</sup> Three possibilities of retention for porcelain bonded to metal can be observed: Van der Waals forces, micromechanical retention, and chemical bonding, with chemical bonding being the main determinant of union, as characterized by the direct transfer of electrons between the oxygen in the vitreous part of the ceramic and oxidation of the metal.<sup>5,6</sup>

In an endeavor to improve the bond strength between the metal and ceramic, some surface treatments have been studied, with the goal of increasing the wettability of the metal by porcelain, and also to control the formation of a thin layer of oxides. Among the main treatments, the following are outstanding: preoxidation of the metal before porcelain application, application of bonding agents, airborne particle abrasion, degasification, heat treatment, and mechanical retention with carbide burs and diamond mounted tips,<sup>7-14</sup> however, no standardization was found in the literature with regards to the surface treatment of metal, mainly Co-Cr, before the application of ceramic materials. Therefore, this study analyzed the influence of two types of treatment and esthetic ceramic materials on the durability of shear bond strength between metal and ceramic. The null hypothesis was that the bond strength would be similar between the groups, irrespective of the types of surface treatments and ceramics.

## **Materials and methods**

Two types of ceramics, StarLight Ceram and Duceram Kiss, indicated to be used in combination with Co-Cr alloy were used in the current experiment. Brand names, type, manufacturers, and batch numbers of the ceramics and Co-Cr alloy used in the present study are presented in Table 1.

#### **Fabrication of metallic frameworks**

Cylindrical acrylic templates were milled to the final disc shape (thickness: 4 mm, height: 3.7 mm) and used for fabrication of the frameworks (Fig 1A). Wax sprue formers (Horus, Herpo Produtos Dentários Ltd, São Paulo, Brazil) were perpendicularly attached at one end of the template and were connected to a central wax rod with a 5 mm diameter (Wax Wire for Casting Sprues, Dentaurum, Pforzheim, Germany). The assembly was mounted in a silicone ring and poured with investment material (BellaVest SH<sup>®</sup>, Bego, Bremen, Germany), following manufacturer's recommendations. After the investment material set, the silicone ring and the sprue former were separated from the investment mold. Metallic frameworks were cast in Co-Cr alloy (N = 48) in an electrical induction furnace (Rematitan<sup>®</sup> Autocast, Dentaurum) under argon gas. Elimination of sprues and separation of metallic strips were performed using carbide discs at low speed, and air-particle abrasion with Al<sub>2</sub>O<sub>3</sub>

(110  $\mu$ m; 2 bar pressure) was performed. After separation, the dimensions of the metallic cylinders were verified and adjusted with a diamond drill when necessary.

Half the metallic cylinders had their surfaces air abraided with 50  $\mu$ m aluminum oxide (Korox, Bego) at an angle of 45° for 10 seconds from a distance of approximately 2 cm, under 2 bar pressure. The remaining specimens had their metallic surfaces roughened with a cylindrical tungsten bur (Maxi Cut, Edenta, São Paulo, Brazil), coupled with a cutting machine. The metallic cylinders were perpendicularly positioned in relation to the long axis of the tungsten bur during the roughness procedure, using 1 kg of force for 10 seconds. The frameworks were then ultrasonically cleaned in isopropyl alcohol (Vitasonic II, Vita, Bad Säckingen, Germany) for 5 minutes and were allowed to dry at room temperature.

### Application of ceramic layer

The 48 metallic cylinders were divided into four groups, according to the veneering ceramic (StarLight Ceram, Duceram Kiss) and surface treatment [air-particle abrasion with Al<sub>2</sub>O<sub>3</sub> and tungsten drill (W)] (n = 12): Gr1 – StarLight + Al<sub>2</sub>O<sub>3</sub>; Gr2 – StarLight + W; Gr3 – Duceram + Al<sub>2</sub>O<sub>3</sub>; Gr4 – Duceram + W. Two layers of opaque ceramic (thickness: 0.1 mm each) were prepared by homogenously mixing the powder (opaque ceramic) and liquid in a container and applied with a thin brush onto the metallic surface by the same operator, to standardize the opaque layers (Table 2). The thickness of the opaque layer was carefully measured using a digital caliper (Starrett 727, Starrett, Itu, Brazil). Specimens with an opaque layer thinner than  $0.2 \pm 0.02$  mm were not included in the groups, and new specimens were added to ensure there were 12 specimens in each group.

The veneering ceramics (StarLight Ceram, Shade 2M1 and Duceram Kiss, Shade 2M1) were fired onto the Co-Cr cylinders using a polyethylene mold supported by a metal base fixed with screws, having an internal diameter of 4 mm and height of 3.7 mm. Sintering of the veneering ceramics was accomplished in an oven (Vacumat, VITA Zahnfabrik). A second firing was performed to compensate for sintering contraction of the ceramics, until a thickness of 4 mm was achieved (Table 2). The specimens were aged using thermal cycling ( $3000 \times$ , 5 to 55°C, dwelling time: 30 seconds, transfer time: 2 seconds).<sup>15</sup>

Brand name	Ceramic type	Manufacturer	Batch no.
StarLight Ceram	Low-fusing ceramic	DeguDent*	003
Starlight Ceram paste opaque	Opaque	DeguDent*	003
Duceram Kiss	Low-fusing ceramic	DeguDent*	60506
Duceram Kiss paste opaque	Opaque	DeguDent*	60506
Cobalt–Chromium alloy	N/A	DeguDent*	60506
(StarLoy C)			

\*Hanau, Germany.



Figure 1 (A) Final shape and dimensions of the ceramic-alloy specimen, (B) lateral view of the metallic device used to fix the ceramic-alloy specimen during the shear test, (C) application of the force on metal–ceramic interface using a shear metallic device (knife notch).

#### Shear strength test

Table 2 Firing procedures

The shear bond strength tests were performed in a universal testing machine (EMIC, mode DL-1000, Equipments and Systems Ltd., Sao Jose dos Pinhais, Brazil) where the load was applied to the metal–ceramic interface at a constant speed of 0.5 mm/min until fracture (Fig 1B–C). Specimens were analyzed under a stereomicroscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany) at  $30 \times$  magnification, and the image was digitally recorded with a camera (Cybershot, Model DSC S85, Sony, Tokyo, Japan) connected to the microscope to characterize the metal surfaces and the failure modes. Some representative specimens were also observed under scanning electron microscopy (SEM) (JEOL-JSM-T330A, Jeol Ltd, Tokyo, Japan).

Energy Dispersive Spectrometer (EDS) analysis was performed on two specimens from each group to determine the chemical elements present at the zone of interaction, using a scanning electronic microscope (JEOL-JSM-5400, Jeol Ltd) equipped with an energy dispersive X-ray (15 Kv; dwell time: 20% to 30% of saturation) and the INCA Energy program. The data were collected as line profiles across the sample ceramic/metal interface. The accelerating voltage was set at 20 kV, the specimen working distance was 15 mm, and the X- ray detector was set to 5 cm throughout the experiments. One area of the interface was selected for scanning under secondary electron (SE) at  $750 \times$  magnification. The failure types were classified as: (A) adhesive along the interfacial region between the metal and the veneering ceramic; (B) cohesive in the metal; (C) cohesive in the veneering ceramic; and (D) mixture of adhesive failure between the veneering and the metal together with cohesive fracture of the veneering ceramic.

#### Statistical analysis

Statistical analysis was performed using Statistix for Windows (Analytical Software Inc., version 8.0, 2003, Tallahase, FL). The means (MPa) of each group were analyzed using 2-way ANOVA and Tukey's test, with the variables of "veneering ceramic types" and "surface treatment." *p*-values less than 0.05 were considered to be statistically significant in all tests.

## Results

The results of 2-way ANOVA for the experimental conditions are presented in Table 3. The mean bond strength values were significantly affected by the ceramic types (p = 0.03252) and surface treatment types (p = 0.0002). The interaction between

	Starting	Drying	Final	Temperature rate	Holding
Ceramics	temperature (°C)	time (min)	temperature (°C)	of increase (°C/min)	time (min)
StarLight Ceram					
First opaque layer	575	7	980	55	2
Second opaque layer	575	7	950	55	2
First dentine layer	575	6	910	55	1
Second dentine layer	575	4	900	55	1
Duceram Kiss					
First opaque layer	575	7	930	55	2
Second opaque layer	575	7	930	55	2
First dentine layer	575	6	910	55	1
Second dentine layer	575	4	880	55	1

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**Table 3** Results of 2-way ANOVA for the ceramic types, surface treatment conditions, and their interaction, according to shear bond strengthdata (p < 0.05)

Effect	DF	SS	MS	F	р
Ceramic	1	647.90	647.90	4.72	0.0352*
Surface treatment	1	2291.77	2291.77	16.70	0.0002*
Interaction	1	150.06	150.06	1.09	0.3014
Residue	44	6038.46	137.24		
Total	47	9128.19			

\*Statistically significant difference at the level of 5%.

the ceramic types and surface treatment factors were not statistically significant (p = 0.3014).

The results of Tukey's multiple comparison test demonstrated that when the main factor *surface treatment* was analyzed, the Al<sub>2</sub>O<sub>3</sub> treatment (52.53 ± 13.25 MPa) showed significantly higher results than the tungsten drill surface treatment (38.71 ± 11.03 MPa), but this difference was only significant for the StarLight ceramic (Gr1: 57.97 ± 11.34 MPa, Gr2: 40.62 ± 12.96 MPa) (Tukey's test) (Table 4, Fig 2). When only the main factor *ceramic type* was analyzed, regardless the surface treatment factor, the StarLight (49.29 ± 14.85 MPa) presented significantly higher results than Duceram Kiss (41.95 ± 12.18 MPa) (p = 0.0352) (ANOVA 2-way and Tukey's test) (Table 3).

The fracture analysis at  $30 \times$  magnification showed a mixture of adhesive failure between the veneering ceramic and metal together with cohesive fracture of the veneering ceramic (Score D) for all groups (Table 5, Figs 3 and 4). In the SEM analysis of longitudinal sections of the specimens, three regions were detected: (a) metal substrate, (b) metal-ceramic interaction zone, (c) ceramic substrate. It was verified that all the interfaces presented as intact, with good contact and wettability between the ceramic and the Co-Cr surface, without presence of faults or slits, suggesting an appropriate adhesion between the two materials. Representative specimens of each ceramic/Co-Cr system, according the surface treatments and without aging, are shown in Figures 5 to 8. It is also possible to observe that air-particle abrasion with Al<sub>2</sub>O<sub>3</sub> created microretention on the metallic surface using SEM analysis (Figs 5-8), contributing to the higher bond strength for those groups (Gr1, Gr2), whereas the tungsten drill produced macroretention (Gr3, Gr4) less efficiently than the other surface treatment.

 $\label{eq:table_table} \begin{array}{l} \mbox{Table 4} & \mbox{Mean} \pm \mbox{standard} \mbox{deviations of the shear bond values (MPa) for ceramic-Co-Cr and surface treatment combinations \end{array}$ 

Experimental groups	Surface tr	Surface treatment			
	Al <sub>2</sub> O <sub>3</sub>	Tungsten drill			
StarLight Ceram Duceram Kiss	$\begin{array}{l} 57.97 \pm 11.34^{a} \\ 47.09 \pm 13.19^{a,b} \end{array}$	$\begin{array}{c} 40.62 \pm 12.96^{b} \\ 36.80 \pm 8.86^{b} \end{array}$			
$Mean \pm SD$	$52.53\pm13.25$	$38.71 \pm 11.03$			

Same superscript letters indicate no significant differences (Tukey's test,  $\alpha = 0.05$ ).



Figure 2 Means of the shear bond strength values according to the experimental conditions: ceramic and surface treatment.

The following elements were observed in the zone of interaction using EDS analysis: Chromium (Cr), Cobalt (Co), Oxygen (O), Sodium (Na), Potassium (K), Calcium (Ca), Aluminium (Al), Silicon (Si), Vanadium (V), and Tungsten (W) in all four groups. No atomic interaction was observed in the alloy surface after air-particle abrasion, regardless of the ceramic used (Figs 9–12).

The null hypothesis was partially accepted.

## Discussion

The primary requirement for a successful metal–ceramic restoration is the development of a lasting bond between the ceramic and metal alloy. Once this bond is obtained, there is the possibility of introducing stresses to this system during the ceramic sinterization process. An unfavorable distribution of stresses during the cooling process could also result in ceramic fracture; moreover, late fracture could also occur. Fractures or delamination of the esthetic ceramic covering are serious and costly problems in dentistry, causing functional and esthetic inconveniences, both for the patient and the dentist.<sup>16</sup>

The Co-Cr alloy (StarLoy C) used in the current study is characterized as having good mechanical properties, allowing thin structures with a smaller volume of material used;<sup>17</sup> a thermal expansion coefficient close to that of the ceramics used, enabling a reduction in stresses and cracks between the two materials after the ceramic firing; biocompatibility, because there is no Ni or Be in the alloy, as these elements have a

Table 5 Incidence of failure types (%) after shear bond strength test

Experimental groups			
StarLight Ceram/Al <sub>2</sub> O <sub>3</sub>	A(0) B(0) C(0) D(100)		
StarLight Ceram/W	A(0) B(0) C(0) D(100)		
Duceram Kiss/Al <sub>2</sub> O <sub>3</sub>	A(0) B(0) C(0) D(100)		
Duceram Kiss/W	A(0) B(0) C(0) D(100)		

A: Failure between adhesive along the interfacial region between the metal and the veneering ceramic; B: cohesive in the metal; C: cohesive in the veneering ceramic; and D: mixture of adhesive failure between the veneering and the metal, together with cohesive fracture of the veneering ceramic.



**Figure 3** Optical microscopic images of the StarLight Ceram/Co-Cr specimens ( $\times$ 25) after shear bond strength testing, showing ceramic layer remnants on the Co-Cr surface for (A) Al<sub>2</sub>O<sub>3</sub> and (B) tungsten bur specimens.



Figure 4 Optical microscopic images of the Duceram Kiss/Co-Cr specimens ( $\times$ 25) after shear bond strength test showing ceramic remnants on the Co-Cr surface for (A) Al<sub>2</sub>O<sub>3</sub> and (B) tungsten bur specimens.



Figure 5 Representative SEM micrograph ( $\times$ 1000) of the StarLight Ceram/Co-Cr interface after Al<sub>2</sub>O<sub>3</sub> surface treatment. Note the good wettening of the ceramic on the metal and the presence of microretention on the metal surface: (a) ceramic, (b) interaction zone, and (c) metal.



Figure 6 Representative SEM micrograph ( $\times$ 1000) of the Duceram Kiss/Co-Cr interface after Al<sub>2</sub>O<sub>3</sub> surface treatment. Note the good wettening of the ceramic on the metal and the presence of microretentions on the metal surface: (a) ceramic, (b) interaction zone, and (c) metal.



**Figure 7** Representative SEM micrograph (×1000) of the StarLight Ceram/Co-Cr interface after tungsten bur surface treatment. Note the good wettening of the ceramic on the metal and the absence of microretention on the metal surface: (a) ceramic, (b) interaction zone, and (c) metal.



**Figure 8** Representative SEM micrograph (×1000) of the Duceram Kiss/Co-Cr interface after tungsten bur surface treatment. Note the good wettening of the ceramic on the metal and the absence of microretention on the metal surface: (a) ceramic, (b) interaction zone, and (c) metal.





**Figure 10** EDS analysis of a StarLight Ceram/Co-Cr interface after tungsten bur surface treatment.

Figure 11 EDS analysis of a Duceram Kiss/Co-Cr interface after  $Al_2O_3$  surface treatment.

Figure 12 EDS analysis of a Duceram

treatment.

Kiss/Co-Cr interface after tungsten bur surface

carcinogenic and allergenic potential but mechanical properties similar to those of Co-Cr alloys.<sup>18,19</sup> An important factor to consider when one wishes to study the bond between metal and ceramic concerns the mechanical tests to use in the study.

Some authors affirm that there is no methodology capable of measuring purely the shear forces at the bond interface,<sup>20-22</sup> as there is no pure shear load at the metal-ceramic interface. Moreover, there is a discontinuity in the stress generated at the points of initial contact with the ceramic. For Lenz and Kessel,23 in both the three-point bending tests and shear tests, a high concentration of stresses occurred in the areas of initial contact of the application of force. On the other hand, some authors<sup>17,24</sup> consider the shear test adequate for measuring the bond strength between metal infrastructure materials and esthetic ceramic coverings, because this type of test is performed to induce the stress directly at the interface of the studied materials. Hammad and Stein<sup>25</sup> related that most of the time, shear tests could direct stresses directly to the interface and not have the results influenced by the modulus of elasticity of the metal, as in the bending tests.

Another aspect that influences the results of shear tests is the device used for performing the test. For Van Noort et al,<sup>26</sup> the shear device, a knife without a groove, proposed by ISO TR 11406,<sup>27</sup> generates only one single point of contact at the metal–ceramic interface of the specimen, therefore, the stress is concentrated in a small area, resulting in a premature failure at the interface. Because of this, the present study used a knife device with a central groove that covered half the test specimen.<sup>28,29</sup>

The descriptive statistics of the shear stress data (MPa) obtained in the current study, according to the experimental conditions, revealed the following means and standard deviations: G1: 57.97  $\pm$  11.34; G2: 40.62  $\pm$  12.96; G3: 47.09  $\pm$  13.19; and G4: 36.80  $\pm$  8.86. Statistical analysis of the results showed that the null hypothesis was partially accepted, because the airborne-particle abrasion with Al<sub>2</sub>O<sub>3</sub> significantly increased the metal/ceramic bond strength when compared with the use of tungsten burs only for the StarLight Ceram ceramic (p < 0.05). This can be explained by some factors, including the fact that the chemical bond between Co-Cr alloy and Duceram ceramic is weaker than the Co-Cr alloy and StarLight Ceram ceramic and that there is no pure shear load at the metal–ceramic interface, even when using a knife device with a central groove.<sup>26</sup>

In accordance with ISO Standard 9693,<sup>30</sup> an adequate bond between a metal alloy and ceramic occurs when the shear bond strength is higher than 25 MPa, suggesting clinically acceptable values for this study. Similar values were found by Pretti et al<sup>17</sup> and Melo et al,<sup>31</sup> who used the piston-type shear device, which allows a reduction of the stress on a single point at the interface, as with the knife device with groove.

Tungsten burs are used for finishing metal surfaces of Ni-Cr and Co-Cr alloy copings after removing the sprues. SEM analysis determined that the surface treatment with tungsten tips promoted the formation of nonuniform macroretention on the metal surface, which presented as flat surfaces, with no significant increase in the contact surface of the metal generating a less efficient bond between metal and ceramic (Figs 7 and 8). Moreover, the presence of bubbles is observed in the photomicrographs, which could certainly influence the reduction in bond strength of the specimens in which this surface treatment was performed. According to Hofstede et al,<sup>32</sup> these bubbles could be the result of a nonuniform surface of the metal, such as fissures. This nonuniform surface could trap air and contaminant agents, causing a porosity in the porcelain during sinterization. These porosities would result in areas of localized stress that could lead to premature fracture.

Microretention increased the available metal contact surface of the specimens treated with  $Al_2O_3$  airborne-particle abrasion, as verified by SEM (Figs 5 and 6). According to Graham et al,<sup>14</sup> airborne-particle abrasion increases the surface roughness, increasing the retention of the ceramic by micromechanical action, and produces a less irregular surface.

The reasons for the clinical fractures of porcelain in metal–ceramic systems are multifactorial. As in the oral medium, frequently repeated stress can be found during masticatory functions. Moreover, the influence of water and fatigue caused by cyclic loading is considered an important factor in the durability of these restorations.<sup>16</sup> The most frequent failures are due to the presence of cracks inside the ceramics, which could occur due to stress generated by the different heat expansion coefficients between metal and porcelain,<sup>8,17,33,34</sup> technical errors, such as the incorporation of air bubbles during preparation, contributing to weakening and eventual fracture of the ceramic. Lack of support for the ceramic and an incorrect preparation of the abutment teeth are factors that cannot be ignored when mentioning clinical failures.<sup>16,28,35</sup>

Analysis of the fracture type revealed that 100% of the specimens presented mixed fracture: adhesive failure between the metal and ceramic and cohesive failure in the covering ceramic, with the ceramic fragment in contact with the metal always being located in the superior region of the specimen, that is, in the region in contact with the load. The oxide layer of the ceramic surface was detached when examining the ceramic fragments. According to Papazoglou and Brantley,<sup>36</sup> this provides evidence for the excellent bond strength between the metal and ceramic.

Finally, the findings of the current study seem to have important relevance for metal–ceramic bond strength; however, some limitations, such as size and shape of the specimens are not the same as those used clinically, and may not indicate the real clinical performance of these materials. Further in vitro long-term studies using mechanical fatigue tests on teeth restored with metal–ceramic crowns and prospective clinical studies must be conducted for more clinically relevant results.

## Conclusions

Under the conditions of this study, air-particle abrasion with  $Al_2O_3$  improved the shear bond strength between metal and ceramics used.

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