Opaque Layer Firing Temperature and Aging Effect on the Flexural Strength of Ceramic Fused to Cobalt-Chromium Alloy

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Abstract

Purpose: To evaluate the effect of the opaque layer firing temperature and mechanical and thermal cycling on the flexural strength of a ceramic fused to commercial cobalt-chromium alloy (Co-Cr). The hypotheses were that higher opaque layer temperatures increase the metal/ceramic bond strength and that aging reduces the bond strength.
Materials and Methods: Metallic frameworks ($25 \times 3 \times 0.5$ mm^3; ISO 9693) ($N = 60$) were cast in Co-Cr and airborne-particle abraded (Al_2O_3: 150 μm) at the central area of the frameworks (8×3 mm^2) and divided into three groups ($N = 20$), according to the opaque layer firing temperature: Gr1 (control)—900°C; Gr2—950°C; Gr3—1000°C. The opaque ceramic (Opaque, Vita Zahnfabrick, Bad Säckingen, Germany) was applied, and the glass ceramic (Vita Omega 900, Vita Zahnfabrick) was fired onto it (thickness: 1 mm). While half the specimens from each group were randomly tested without aging (water storage: 37°C/24 hours), the other half were mechanically loaded (20,000 cycles; 50 N load; distilled water at 37°C) and thermocycled (3000 cycles; 5°C to 55°C, dwell time: 30 seconds). After the flexural strength test, failure types were noted. The data were analyzed using 2-way ANOVA and Tukey's test ($\alpha = 0.05$).
Results: Gr2 (19.41 ± 5.5 N) and Gr3 (20.6 ± 5 N) presented higher values than Gr1 (13.3 ± 1.6 N) ($p = 0.001$). Mechanical and thermal cycling did not significantly influence the mean flexural strength values ($p > 0.05$). Increasing the opaque layer firing temperature improved the flexural bond strength values ($p < 0.05$). The hypotheses were partially accepted.
Conclusion: Increasing of the opaque layer firing temperature improved the flexural bond strength between ceramic fused to Co-Cr alloy.

The high cost of noble metal alloys has led to the development and increased clinical use of base metal alloys for manufacturing frameworks for fixed and unitary partial prostheses.[1-3] Among the base metals, the most commonly used are nickel-chromium and cobalt-chromium (Co-Cr).[3,4] Nickel-chromium with beryllium alloys can present a toxicity related to the beryllium, as well as an allergenic potential of the nickel,[4,5] leading to the increasing use of Co-Cr alloys due to their compatibility.[5] Moreover, the nonprecious alloys present excellent mechanical properties,[1-3] such as resistance to permanent deformation and high modulus of elasticity, giving these alloys the advantage of obtaining a thin and rigid framework required for partial fixed prostheses.[3,4]

Metal/ceramic bond failures are common after cementation of fixed partial prostheses with frameworks made of alternative alloys. It is possible that those failures occur during the laboratory phase of the restoration manufacturing and are manifested clinically with the stresses applied during or after the cementation procedure.[6] The difficulty in controlling the formation of an oxide layer on the metal surface at high temperatures is one of the main factors responsible for these failures, negatively affecting the metal/ceramic bond strength.[7-9]

Small changes in laboratory procedures may have a significant impact on the metal/ceramic bond; therefore, strictly following the details and precision in the manufacturing process is essential to the success of restorations;[3] however, some changes in laboratory procedures are suggested by some authors to increase the bond strength between the materials, such as: the use of intermediate bonding agents,[10,11] different ceramic firing temperatures,[12] changes in the ceramic cooling rate,[6] the use of different ceramic firing environments,[3,13] increasing the number of ceramic firings,[14,15] changing the metal treatment,[16,17] the use of an opaque layer,[18] and increasing the firing temperature of the opaque layer.[3,8,19]

The purpose of this study was to evaluate the influence of three opaque layer firing temperatures and mechanical and thermal cycling on the metal/ceramic bond when submitted to the 3-point flexural strength test. The hypotheses were that increasing the opaque layer temperature improved the metal/ceramic bond strength and that aging reduced the flexural strength.

Materials and methods

Fabrication of metallic frameworks

Rectangular acrylic templates (27 × 3 × 0.5 mm^3) were used for the fabrication of the frameworks.[3] Wax sprues (Horus, Herpo Produtos Dentários Ltd, São Paulo, Brazil) were perpendicularly attached at one end of the template and 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 T, Bego, Bremen, Germany). After the investment material was set, the silicone ring and the sprue former were separated from the investment mold. The metallic frameworks were cast in Co-Cr (Wirobond C, Bego) (N = 60) in an electrical induction furnace (Fornax GEU, Bego) under argon gas. The sprues were eliminated, and the metallic strips separated with the aid of carbide discs at low speed.

After removal from the investment material, the margins of the frameworks were trimmed to the final dimensions of 25 × 3 × 0.5 mm[33,20] with the measurements controlled using a digital paquimeter with a precision of 0.01 mm (Model Starrett 727, Starrett, Itu, Brazil). The surfaces of the specimens to receive the ceramics were airborne-particle abraded with 150-μm aluminum oxide (Korox, Bego) at an angle of 45° for 10 seconds from a distance of approximately 2 cm, under 2 bar pressure. The frameworks were then ultrasonically cleaned in isopropyl alcohol (Vitasonic II, Vita Zahnfabrick, Bad Säckingen, Germany) for 10 minutes and dried at room temperature.

Application of ceramic layer

An area of 8 × 3 mm^2 was initially marked on the Co-Cr frameworks with a graphite pencil. Using a metallic device and a brush, a thin opaque layer (Wash Opaque WO 9000, Vita Zahnfabrick, #7268) was applied on the framework area marked. The opaque was applied on the bonder by pulverization of powder (opaque ceramic) and liquid, and homogenized in a container connected to a dispenser. The thickness of the ceramic layer (Vita Omega 900, Vita Zahnfabrick, #5475) corresponding to dentin ceramic (1 mm) was standardized by positioning the frameworks in a metallic template (Fig 1).

After removal from the assembly, the ceramic was fired (Vacumat 40, Vita Zahnfabrick) (Table 1). Due to shrinkage, a second layer was applied, and the specimens were submitted to a final glaze firing. The opaque layer firing temperature varied according to the groups: Gr1—900°C, Gr2—950°C, Gr3—1000°C (Table 2).

Mechanical and thermal cycling

Twenty specimens for each opaque firing/Co-Cr combination were randomly divided into two subgroups: one subjected to mechanical and thermal cycling and the other stored in distilled water for 24 hours at 37°C (control group) prior to flexural strength test. Mechanical cycling of the specimens was carried out in a mechanical cycling machine (custom made, São Paulo State University, Dental School, UNESP, São José dos Campos, Brazil) developed to simulate the mechanical forces generated during the chewing cycle. The device used for this test was composed of two bases, 2 cm apart from each other, on which cylinders (radius: 1.0 mm), were placed to allow positioning of the specimens parallel to the ground and perpendicular to the axial load. An upper rod with a 1-mm diameter tip was fixed on the plier that induced a 50 N load 20,000 times with a frequency of 1 cycle/sec. The testing device was placed on a machine base

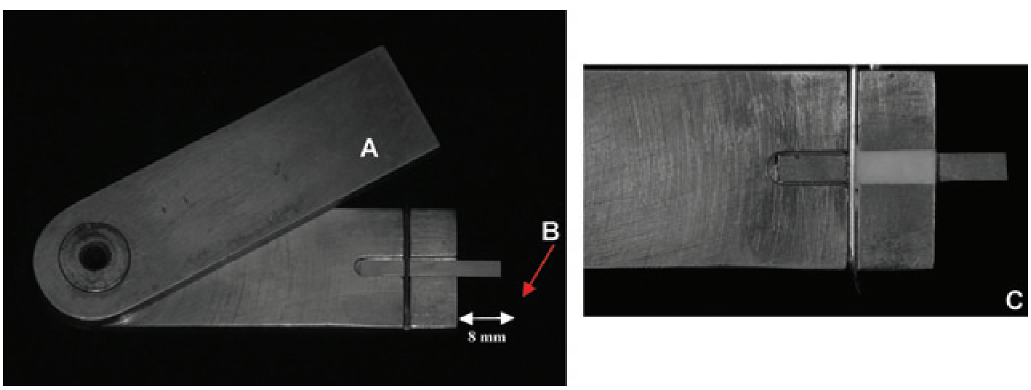

Figure 1 (A) Metallic device used to apply the opaque/dentin ceramics at the cross-section dimensions according to the ISO 9396, (B) metallic bar positioned on device before opaque layer application, and (C) after ceramic layer application.

Table 1 Firing procedures of the dental ceramic tested

Ceramic Vita Omega 900	Starting temperature (°C)	Drying time (min)	Final temperature (°C)	Temperature rate of increase (°C/min)	Holding time (min)
Opaque	600	4	900	75	1
1st dentine layer	600	4	900	75	1
2nd dentine layer	600	6	900	50	1

Table 2 Firing procedures of the opaque ceramic for all groups

Ceramic Vita Omega 900	Starting temperature (°C)	Drying time (min)	Final temperature (°C)	Temperature rate of increase (°C/min)	Holding time (min)
Gr1 (control)	600	4	900	75	1
Gr2	600	4	950	88	1
Gr3	600	6	1.000	100	1

containing a thermostat to allow testing in an aqueous medium at a constant temperature of 37°C.

The specimens were then thermocycled for 3000 cycles between 5°C and 55°C in deionized water (Nova Etica, São Paulo, Brazil). The dwell time at each temperature was 10 seconds, and the transfer time from one bath to the other was 5 seconds.

Flexural strength test

The flexural tests were performed in a universal testing machine (Instron 4301, Instron Corp., Norwood, MA), with the load applied at a constant speed of 1.5 mm/min until fracture. The load that led to the initial separation of materials was obtained in kgf and converted to N, by means of the following equation:

$$N = F(Kgf) \times 9.8 m/sec.$$

Fracture analysis

The specimens were analyzed under a stereomicroscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany) under a magnification of 30 ×, 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. The failure types were classified as: (1) adhesive along the interfacial region between the opaque ceramic and the interaction zone, (2) inside the interaction zone, and (3) cohesive along the interfacial region between the metal and the interaction zone.[21]

Analysis of the metal-ceramic interface

In addition to the experimental groups, two specimens of each group were made. These specimens were not subjected to the bending test, but were examined under SEM. Before examination, the specimens were embedded in autopolymerizing acrylic resin, longitudinally sectioned, finished with 220 to 1200 grit and polished with diamond paste (6, 3, and 0.25 μm) and felt under water coolant irrigation (POLI PAN-2/Panambra, Sao Paulo, Brazil).

The morphological analysis of the metal-ceramic interface for each group was carried out with a scanning electron microscope (SEM) (LEO 435 VPI/LEO-Zeiss, Tokyo, Japan). The data were collected as a line profile across the ceramic-metal interface of the specimens. One area of the interface was selected for scanning under secondary electron mode at 150 × magnification.

Statistical analysis

The means of each group were analyzed by 2-way ANOVA, with flexural strength test as the dependent variable and the opaque firing temperature-metal combinations and fatigue conditions as the independent factors. P-values less than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made by Tukey's adjustment test.

Results

The results of the 2-way ANOVA for the experimental conditions are presented in Table 3. The interaction between the opaque firing temperature and the cycling factors was not statistically significant ($p = 0.1697$) (ANOVA, Tukey's test), indicating that the metal/ceramic bond strength (N) obtained for the different opaque firing temperatures were constant for both the absence and presence of cycling (Fig 2). Mechanical and thermal cycling did not significantly decrease the mean

Table 3 Results of 2-way ANOVA for the opaque firing temperature, cycling fatigue conditions, and the interaction terms according to flexural strength data

Effect	DF	SS	MS	F	*p*
Temperature	2	611.62	305.810	16.29	0.001*
Cycling	1	14.78	14.781	0.79	0.3789
Interaction	2	68.84	34.422	1.83	0.1697
Residue	54	1013.92	18.776		
Total	59	1709.16			

*Statistically significant difference at the level of $p < 0.05$. DF: Degrees of freedom; SS: Sum of squares; MS: Mean ratio square; F: Probability F; P: p-value.

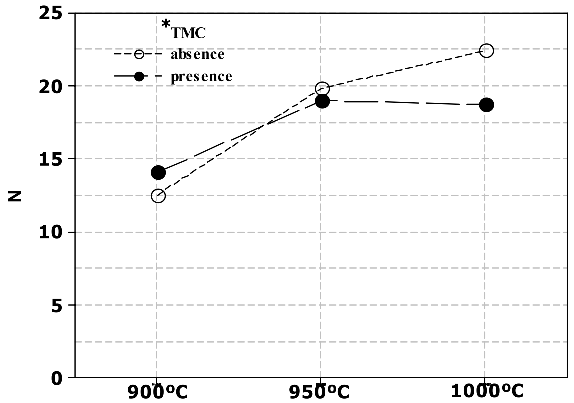

Figure 2 Means of the flexural strength values (N) according to the experimental conditions established by the variables: temperature and aging. *Thermal-mechanical cycling.

flexural strength values ($p < 0.05$) for all group combinations (Gr1 = 14.1 ± 1.7 N; Gr2 = 19 ± 6.7 N; Gr3 = 18.74 ± 3.1 N) when compared to the control group, in which the tests were performed after storage in water at 37°C for 24 hours (Gr1 = 12.52 ± 1 N; Gr2 = 19.8 ± 4.2 N; Gr3 = 22.43 ± 5.9 N) (Table 4).

The results of Tukey's multiple comparison test established that when the main factor of temperature was analyzed individually, higher values ($p < 0.05$) were obtained for the groups with higher opaque layer temperature (950 and 1000°C). Gr2 and Gr3 presented similar results for their control and experimental groups ($p > 0.05$) (Table 4).

In the SEM analysis, complementary to the flexural strength test, three regions were detected from the longitudinal sections of the specimens: (1) metal substrate; (2) metal/ceramic interaction zone; and (3) ceramic substrate. All the interfaces were intact, with a good contact and wettability between the ceramic and the Co-Cr, without the presence of faults or slits, suggesting an appropriate adhesion between the two materials. The representative specimens for each metal-ceramic condition without aging are shown in Figures 3 to 5.

Stereomicroscope images at 30× magnification showed exclusively adhesive failures at the opaque ceramic/Co-Cr interfacial zone for Gr1 (900°C), with no presence of ceramic on the metallic surface but with a visible dark oxide layer in all

Table 4 The mean (± standard deviations) flexural strength values (N) for opaque firing temperature-Co-Cr combinations with and without and mechanical- and thermal-cycling conditions. (Tukey's test, $\alpha = 0.05$)

Experimental groups	Mechanical and thermal cycling		Mean (SD)
	Without	With	
Gr1—900°C	12.52 ± 1*	14.1 ± 1.7*	13.3 ± 1.6
Gr2—950°C	19.8 ± 4.2*	19 ± 6.7*	19.41 ± 5.5
Gr3—1.000°C	22.43 ± 5.9*	18.74 ± 3.1*	20.6 ± 5
Mean (SD)	18.25 ± 4.5	17.28 ± 3.8	

*Means followed by identical letters do not differ statistically.

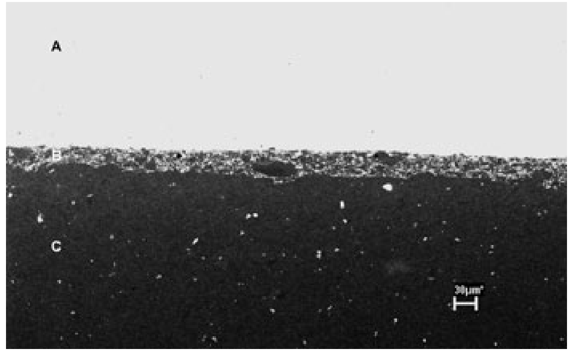

Figure 3 Representative SEM micrograph (×150) of the Vita Omega 900 (Vita Zahnfabrick, Bad Säckingen, Germany) ceramic/Co-Cr surface before aging, at 900°C opaque firing temperature. Note the good wettening of the ceramic on the metal: (A) metal, (B) interaction zone, and (C) ceramic.

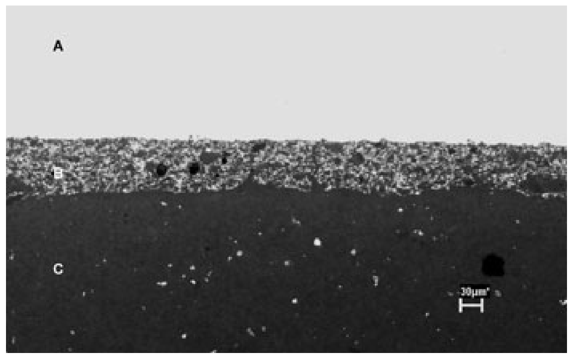

Figure 4 Representative SEM micrograph (×150) of the Vita Omega 900 (Vita Zahnfabrick, Bad Säckingen, Germany) ceramic/Co-Cr surface before aging, at 950°C opaque firing temperature. Note the good wettening of the ceramic on the metal: (A) metal, (B) interaction zone, and (C) ceramic.

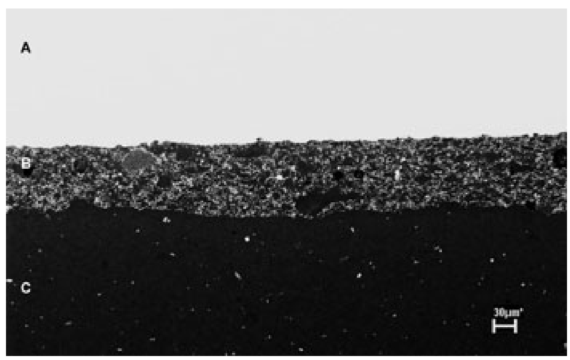

Figure 5 Representative SEM micrograph (×150) of the Vita Omega 900 (Vita Zahnfabrick, Bad Säckingen, Germany) ceramic/Co-Cr surface before aging, at 1000°C opaque firing temperature. Note the good wettening of the ceramic on the metal: (A) metal, (B) interaction zone, and (C) ceramic.

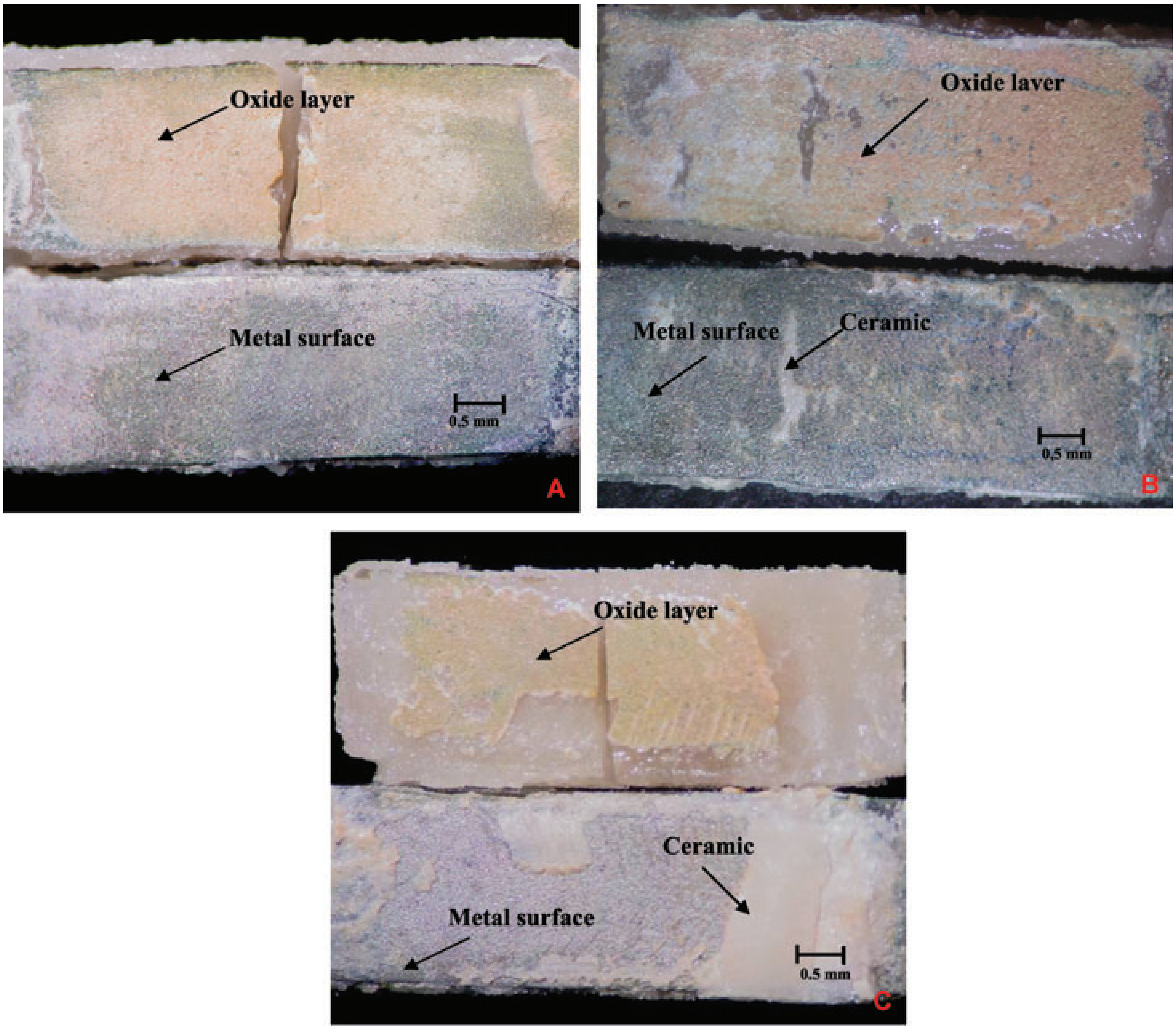

Figure 6 Optical microscopic images of the Vita Omega 900 (Vita Zahnfabrick, Bad Säckingen, Germany)/Co-Cr specimen (× 30) after flexural strength test (A) showing neither ceramic nor oxide layer remnants on the Co-Cr surface at Gr1 (900°C), (B) opaque layer remnants on Co-Cr surface at groups Gr2, and (C) Gr3.

experimental specimens. Cohesive failures at the opaque layer were observed in all specimens for the other groups (950°C and 1000°C). The representative images of metal and ceramic substrates after flexural strength testing with and without aging are illustrated in Figure 6.

The hypotheses were partially accepted.

Discussion

This study evaluated the bond strength between a Co-Cr dental alloy (Wirobond C) and feldspathic porcelain (Vita Omega 900, Vita Zahnfabrick) due to the viability of using Co-Cr alloy for the manufacturing of frameworks for fixed and unitary partial prostheses and because of the few studies on this subject reported in literature. Since there is no agreement in the literature with regard to the most indicated test to evaluate the bond strength between these two materials, many in vitro studies have been proposed to measure the metal/ceramic bond strength, including the traction test,[7,22] shear bond strength,[6,12,15,17] the 3-point flexural bond strength,[1,3,18,23,24] and the 4-point flexural bond strength.[16,25] On the other hand, Della Bona and Van Noort analyzed the shear bond strength and observed that this kind of test created arch-shaped cohesive fractures in all specimens.[26] These fractures occur because of the highly nonuniform tension distribution on the interface of the materials; however, the flexural bond strength test better simulates clinical conditions, since the specimens are under compression, traction, and shear bond strength simultaneously.[27] Moreover, the flexural bond strength test (3-points) is recommended by the International Organization for Standardization[20] to evaluate the metal/ceramic bond strength. For this reason, the 3-point flexural bond strength test, which was proven effective in evaluating the metal/ceramic bond strength using three firing temperatures of the opaque layer, was chosen for this study.

The results of this study can be understood by the analyses of the firing temperature of the opaque layer and by the absence or presence of thermal and mechanical cycling. The effect of this interaction on the metal/ceramic bond strength was not significant.

Three opaque layer firing temperatures were used in this study: 900°C, recommended by the manufacturer, and two experimental temperatures of 950°C and 1000°C. The results of the bond strength testing observed in the groups in which the temperatures were higher than the temperature recommended by the manufacturer, that is, 950°C (Gr2 = 19.41 ± 5.50 N) and 1000°C (Gr3 = 20.6 ± 5 N) were statistically higher than the results obtained by the 900°C group. The technique of increasing this temperature is based on the hypothesis that there is an increase in electron transference between the glass and metal oxides,[28] suggesting an increase in the metal/ceramic bond strength. Wight et al[13] and Hammad et al[19] evaluated the effect of an increasing opaque layer firing temperature, using temperatures (26°C, 18°C) higher than the one recommended by the manufacturer. They concluded that the increased temperature improved the metal/ceramic bond strength, which corroborates this study. This study revealed that the results of the control group without aging and even after mechanical and thermal cycling were higher than the recommended minimum value of 5.625 N, established by DIN 13.927.[29]

Mechanical and thermal cycling simulates the clinical use of the materials to some extent. Most in vitro experiments are performed using static mechanical tests that do not address the aggressive oral environment. The oral environment is able to induce physicochemical alterations of dental materials. Temperature changes provide conditions for degradation of the bond strength in an aqueous environment[30] while also encouraging mechanical fatigue of the materials themselves or their interfaces triggered by the repeated incidence of chewing loads.[31,32] Thermocycling induces repeated stress at the metal/ceramic interface and weakens the bond between the two components.[33-35] Similarly, Tróia Jr et al[33] suggested that periods of extended immersion time in each bath might produce higher tension at the metal/ceramic interface.

The results of this study showed that the absence or presence of thermal and mechanical cycling did not significantly interfere in the metal/ceramic bond strength; however, a decrease in the bond strength of groups Gr2 and Gr3 was observed, G2.1 and G3.1, not submitted to thermal and mechanical cycling, presented mean bond strength values of 19.84 ± 4.24 and 22.43 ± 5.94 (N), respectively; while groups G2.2 and G3.2, submitted to thermal and mechanical cycling, presented values of 18.99 ± 6.75 and 18.74 ± 3.14 (N), respectively. Surprisingly, there was an increase in bond strength for the control group G1 (900°C) when the specimens were submitted to thermal and mechanical cycling. The bond strength of group G1.1 (noncycled) presented a mean value of 12.52 ± 1.07 N, while group G1.2 (cycled) obtained a mean value of 14.08 ± 1.69 N. In accordance with this study, Tróia Jr et al[33] investigated the influence of thermocycling on the metal/ceramic bond strength and did not find any influence on adhesion of ceramic onto this metal.

This situation of higher bond strengths between basic metals and ceramic is shown when the fracture occurs inside the ceramic and not on its interface.[13,36,37] In this study, after the specimens were evaluated with a stereomicroscope, the presence of an opaque layer and veneering ceramic was observed on the metal surface and eruptions of the oxide layer were observed on the ceramic surface for the experimental groups of 950°C and 1000°C, while in the 900°C group, an oxide layer along all the ceramic surface and the absence of opaque and ceramic layers over the metal surface were observed (Fig 6). These results corroborate the findings of Wight et al[13] and Hammad et al[19] in which the metal/ceramic bond strength was improved with an increased opaque layer firing temperature, fractures inside the ceramic were observed in all specimens fired at higher temperatures than the one recommended by the manufacturer, while the specimens fired according to the temperature recommended by the manufacturer presented fractures only on the interface. The stereomicroscopic images suggest that increasing the opaque firing temperatures allows a greater bond flexural strength between the Wash opaque and Co-Cr alloy.

Conclusion

Based on these results, mechanical and thermal cycling did not significantly influence the flexural bond strength values for all opaque firing temperature/Co-Cr combinations tested when compared to control groups and that increased opaque layer firing temperatures significantly increased the flexural bond strength values.

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