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Effect of Airborne-Particle Abrasion and Mechanico-Thermal Cycling on the Flexural Strength of Glass Ceramic Fused to Gold or Cobalt–Chromium Alloy

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Keywords
Dental materials; airborne-particle abrasion; mechanical and thermal-cycling; chromium alloy; gold alloy; surface treatment; metal/ceramic joint.

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Abstract

Purpose: To evaluate the effect of airborne-particle abrasion and mechanico-thermal cycling on the flexural strength of a ceramic fused to cobalt–chromium alloy or gold alloy.
Materials and Methods: Metallic bars (n = 120) were made (25 mm × 3 mm × 0.5 mm): 60 with gold alloy and 60 with Co–Cr. At the central area of the bars (8 mm × 3 mm), a layer of opaque ceramic and then two layers of glass ceramic (Vita VM13, Vita Zahnfabrick) were fired onto it (thickness: 1 mm). Ten specimens from each alloy group were randomly allocated to a surface treatment [(tungsten bur or air-particle abrasion (APA) with Al_2O_3 at 10 mm or 20 mm away)] and mechanico-thermal cycling (no cycling or mechanically loaded 20,000 cycles; 10 N distilled water at 37°C and then thermocycled 3000 cycles; 5°C to 55°C, dwell time 30 seconds) combination. Those specimens that did not undergo mechanico-thermal cycling were stored in water (37°C) for 24 hours. Bond strength was measured using a three-point bend test, according to ISO 9693. After the flexural strength test, failure types were noted. The data were analyzed using three factor-ANOVA and Tukey's test ($\alpha = 0.05$).
Results: There were no significant differences between the flexural bond strength of gold and Co–Cr groups (42.64 ± 8.25 and 43.39 ± 10.89 MPa, respectively). APA 10 and 20 mm away surface treatment (45.86 ± 9.31 and 46.38 ± 8.89 MPa, respectively) had similar mean flexural strength values, and both had significantly higher bond strength than tungsten bur treatment (36.81 ± 7.60 MPa). Mechanico-thermal cycling decreased the mean flexural strength values significantly for all six alloy-surface treatment combinations tested when compared to the control groups. The failure type was adhesive in the metal/ceramic interface for specimens surface treated only with the tungsten bur, and mixed for specimens surface treated with APA 10 and 20 mm.
Conclusions: Considering the levels adopted in this study, the alloy did not affect the bond strength; APA with Al_2O_3 at 10 and 20 mm improved the flexural bond strength between ceramics and alloys used, and the mechanico-thermal cycling of metal-ceramic specimens resulted in a decrease of bond strength.

The longevity of metal-ceramic restorations depends on the bond strength between the ceramic and the metallic infrastructure. Noble metallic alloys are used to build infrastructures due to their excellent biocompatibility and good mechanical properties with excellent bonding to the covering ceramic.[1] However, the increase in the cost of noble metallic alloys in the 1970s led to the development and increasing clinical use of basic metal alloys to make crown and fixed partial prosthesis infrastructures.[2,3]

Basic alloys present beneficial mechanical properties, such as resistance to permanent deformation and high modulus of elasticity, so that copings made from these alloys have little thickness and adequate rigidity for extensive fixed partial prostheses.[3,4] However, controlling the formation of a metal oxide at high temperatures is difficult with basic alloys, thereby decreasing the efficiency of the metal/ceramic bond.[5,6] Several surface treatments have been studied: degasification,[7] use of intermediate bonding agents,[8] different ceramic firing temperatures,[9] alteration of the ceramic cooling rate,[10] use of different ceramic firing environments,[3,11] increase of the number of ceramic firings,[12] use of the opaque layer,[13] increase of the opaque layer's firing temperature,[11,14,15] use of air-particle abrasion (APA) with Al_2O_3,[14,16–20] and mechanical retention with carbide burs and diamond tips.[11,16,17,20]

APA with Al_2O_3 is used to clean the metal surface to produce a micro-retentive roughness by increasing the surface area.[18,21] However, no standardization was found in the literature with regards to the distances of APA during surface treatment of metal, before the application of ceramic materials. The distances between the sandblaster nozzle and the surface of the metal ranged from 1 to 10 cm.[1,18]

When dental restorations are cemented and exposed to the oral environment, several factors may limit their service life, since dental materials may undergo physicochemical alterations. The incidence of repeated forces during chewing results in stress concentration, and thermal variations induce fatigue of the materials themselves and/or the interface between them.[22] Thus, some authors have proposed several testing methodologies such as thermal or mechanical or mechanico-thermal cycling procedures to simulate the oral conditions prior to mechanical testing.[20,22]

The goal of this study was to evaluate in vitro the influence of APA and mechanico-thermal cycling on the bond strength of ceramic to a gold alloy or cobalt–chromium alloy as measured by the three-point bend test.

Materials and methods

Fabrication of metallic bars

Rectangular acrylic templates (27 mm × 3 mm × 0.5 mm) were used for the fabrication of the bars. Wax sprues (Horus, Herpo Produtos Dentários Ltd, São Paulo, Brazil) were perpendicularly attached at one end of the template and connected to a 5-mm-diameter central wax rod (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 bars were cast in Co–Cr (Wirobond® C, Bego) (N = 60) or in gold alloy (Olympia-Jelenko, Heraeus Kulzer, Hanau, Germany) (N = 60) in an electrical induction furnace (Fornax GEU®, Bego) under argon gas. The sprues were eliminated and the metallic strips separated with the help of carbide discs at low speed. After removal from the investment material, the margins of the bars were trimmed to the final dimensions of 25 mm × 3 mm × 0.5 mm,[23] with measurements controlled using a 0.01 mm precision digital caliper (Model Starrett 727, Starrett, Itu, Brazil).

Table 1 Experimental groups, alloys, and surface treatment (n = 20)

Groups	Alloy	Surface treatment
G1		Surface grinding with tungsten bur
G2	Gold	Surface grinding with tungsten bur/ APA with Al_2O_3 (10 mm distance)
G3		Surface grinding with tungsten bur/ APA with Al_2O_3 (20 mm distance)
G4		Surface grinding with tungsten bur
G5	Co–Cr	Surface grinding with tungsten bur/ APA with Al_2O_3 (10 mm distance)
G6		Surface grinding with tungsten bur/ APA with Al_2O_3 (20 mm distance)

One of the sides of the metallic bars was randomly selected and had its metallic surface roughened with a cylindrical aluminum oxide white stone (Shofu, Menlo Park, NJ) and a tungsten bur (Edenta 5720.040, Labordental, Sao Paulo, Brazil) in one direction parallel to the long axis of the metallic bars, following VM13 ceramic manufacturer's instructions. Then, the metallic bars were divided into six groups (n = 20), according to the alloy and metal surface treatment (Table 1).

APA with Al_2O_3 was standardized through a specially developed device, which maintained the metallic bars at a previously selected distance (10 or 20 mm) and at a 90° angle between the sandblaster nozzle and the metal bar (Fig 1). The metallic bars were then ultrasonically cleaned in isopropyl alcohol (Vitasonic II, Vita Zahnfabrik, Bad Säckingen, Germany) for 10 minutes and dried at room temperature.

Ceramic layer application

Prior to opaque application, only gold bars were oxidized without vacuum, from 650 to 1010°C, using a porcelain furnace (Vacumat 40, Vita Zahnfabrik). An area of 8 mm × 3 mm was initially marked on the Co–Cr and the gold bars with a graphite pencil. Using a metallic device and a brush, a thin opaque layer (Wash Opaque VM13, Vita Zahnfabrik, #15790) was applied on the framework's marked area. The opaque was applied

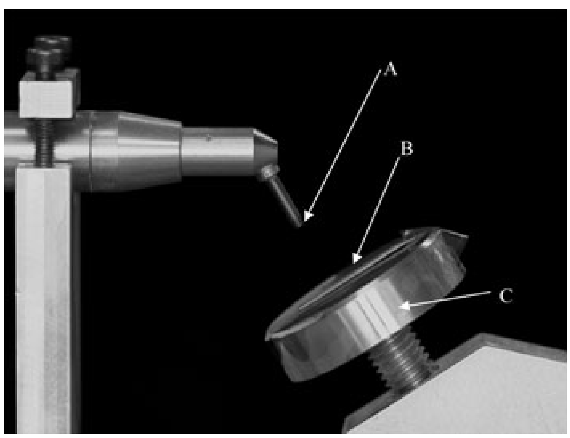

Figure 1 (A) Sandblaster nozzle; (B) metal bar; (C) sliding table.

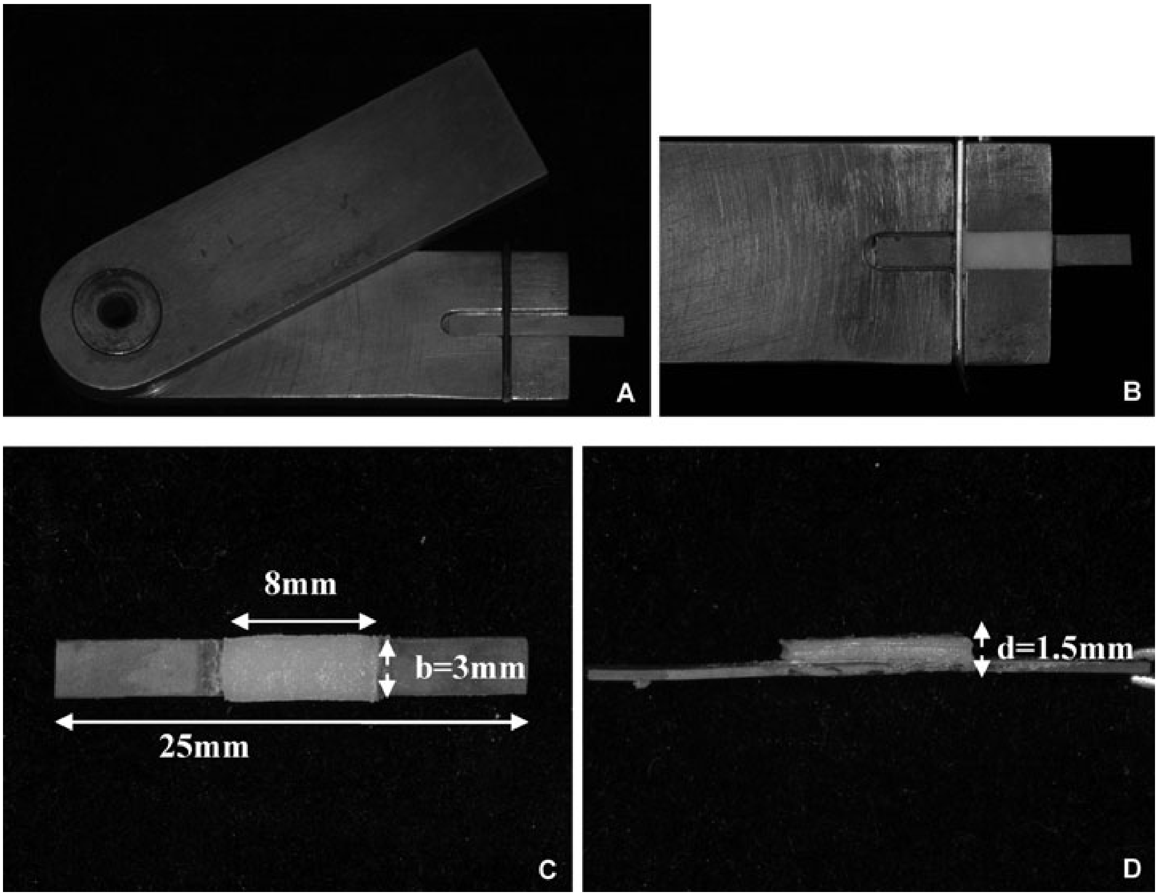

Figure 2 (A) Metallic device used to apply the opaque/dentin ceramics at the cross-section dimensions according to ISO 9396, and metallic bar positioned on device before opaque layer application, (B) After ceramic layer application, (C) Final shape and dimensions of the ceramic–alloy specimen, and (D) Final thickness of the ceramic–alloy specimen (metal = 0.5 mm, ceramic = 1.0 mm).

on the bonder by powder pulverization (opaque ceramic) and liquid, and homogenized in a container connected to a dispenser. The thickness of the ceramic layer (2M2, Vita VM13, Vita Zahnfabrik, #10770) corresponding to dentin ceramic (1 mm) was standardized by positioning the bars in a metallic template (Fig 2).

After removal from the assembly, the ceramic was fired. Due to shrinkage, a second layer was applied, and the specimens were submitted to a final glaze firing (Table 2).

Mechanical and thermal cycling

Sixty specimens of each surface treatment-Gold or surface treatment-Co–Cr combinations 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 a flexural strength test. Mechanical cycling for the specimens was performed in a mechanical stress simulator (Model ER-11000, ERIOS, Sao Paulo, Brazil). Twenty thousand cycles were performed, with a 1 cycle/sec frequency and constant load of 10 N.[22] During cycling, the bars remained immersed in distilled water at a controlled temperature of 37°C. Specimens were then thermocycled for 3000 cycles between 5 ± 1 °C and 55 ± 1°C in deionized water (Nova Etica, Sao Paulo, Brazil). The dwell time at each temperature was 10 seconds, and the transfer time from one bath to the other was 5 seconds. Those specimens stored in distilled water were tested without mechano-thermal cycling at the end of the 24-hour period.

Flexural strength test

The flexural tests were performed in a universal testing machine (DL-1000, EMIC, Curitiba, Brazil), with the load applied at a constant 1.5 mm/min speed until fracture (Fig 3). The load leading to the initial separation of materials was obtained in kilogram force (kgf) and converted to Newtons (N), for the calculation of the flexural strength according to the following equation:

$$\text{flexural strength (MPa)} = \frac{3Pl}{2bd^2}$$

where P was the maximum load upon fracture (N), l the span distance (mm), b the width, and d the thickness of the specimen (mm).

Table 2 Firing procedures of the ceramic used

Ceramic Vita VM13	Starting temperature (°C)	Predrying time (min)	Heating time (min)	Heating temperature (°C)	End temperature (°C)	Hold time for temperature (min)	Hold time for vaccum (min)
Wash Opaque	500	2.0	5.12	75	890	2.0	5.12
Opaque	500	4.0	5.12	75	890	2.0	5.12
1st dentine	500	6.0	6.55	55	880	1.0	6.55
2nd dentine	500	6.0	6.44	55	870	1.0	6.44

Fracture analysis

The specimens were analyzed by visual inspection, and representative images were digitally recorded with a camera (Cybershot, Model DSC S85, Sony, Tokyo, Japan) connected to the stereomicroscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany) to characterize the metal surfaces and the failure modes, under 30× magnification. The failure types were classified as adhesive (along the interfacial region between the opaque ceramic and the metal, cohesive (inside the metal), cohesive (inside the ceramic), or mixture of adhesive failure between the opaque ceramic and the metal with cohesive fracture of the ceramic.

Statistical analysis

Statistical analysis was performed using Minitab version 14.12, 2004 (Minitab, Inc., State College, PA) and Statistix for Windows (Analytical Software Inc., Version 8.0, 2003, Tallahassee, FL). A three-factor ANOVA was used to assess the effect of the type of alloy, surface treatment, and mechanico-thermal cycling on the bond strength. Tukey's test was used for multiple comparisons. The level of significance was set at 5%. The assumptions of the ANOVA were verified prior to analysis: the residuals were normally distributed (statistics, Anderson-Darling = 0.448; p-value = 0.275), and the plot of the residuals against predicted values indicated homogeneity of variance.

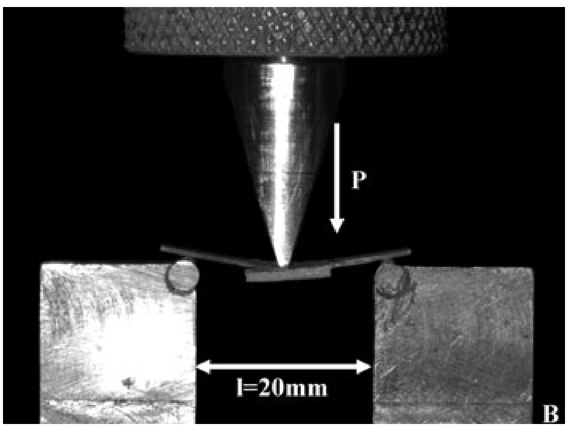

Figure 3 Application of force on the metal until separation of the ceramic from the metal surface.

Results

Table 3 presents the means and standard deviations (SD) for all experimental conditions. Results of the three-way ANOVA for the experimental conditions are presented in Table 4. There were no statistically significant interactions between the explanatory variables, indicating that the pattern of bond strength was not influenced by combinations of factors. There was a statistically significant difference among the average bond strengths for surface treatment and mechanico-thermal cycling but not for the type of alloy. When considering the surface treatment factor, APA 10 and 20 mm surface treatments, had similar mean flexural strength values (APA 10 mm vs APA 20 mm, $p = 0.953382$), and air-abrasion surface treatments had significantly higher average bond strengths than surface treatment with tungsten bur (APA 10 mm vs tungsten, $p = 0.000117$; APA 20 mm vs tungsten, $p = 0.000115$) (Table 5).

Regarding mechanico-thermal cycling, the cycled groups (means and standard deviations: cycled groups = 39.02 ± 9.65 MPa) had a mean flexural bond strength value significantly lower ($p = 0.001$) than the non-cycled groups (means and standard deviations: non-cycled groups = 47.01 ± 7.84 MPa). Homogeneity of variance was also verified by Bartlett test: the residuals were normally distributed (surface treatment factor: statistic $\chi^2 = 1.68$; df = 2; p-value = 0.431; mechanico-thermal cycling factor: statistic $\chi^2 = 2.53$; df = 1; p-value = 0.111).

On visual inspection, groups 1 and 4 (those treated only with tungsten burs, that is, did not receive APA with Al_2O_3) showed primarily adhesive failures along the metal/ceramic interface without visual presence of ceramic on the metallic surface of metal. A visible dark oxide layer was observed on all specimens. All specimens that received APA with Al_2O_3 exhibited an opaque layer and ceramic on the surface of metal, suggesting a mixture of adhesive failure between the opaque ceramic and the metal with cohesive fracture of the ceramic (Table 6). Stereomicroscope images representing the surface of the metal and of the ceramic of all groups after the three-point flexural strength test are illustrated in Figures 4 and 5.

Discussion

This study evaluated the flexural bond strength of a feldspathic ceramic (Vita VM13) to a gold alloy (Olympia-Jelenko) or to a cobalt–chromium alloy (Wirobond C) and found no significant difference between the studied alloys. This study agrees with recent studies.[13,24] Jóias et al[24] compared the shear bond strength of a feldspathic ceramic (Vita Omega 900) to five Co–Cr alloys and a gold alloy. They found that the bond strength between

Table 3 Flexural strength values (MPa) for all experimental conditions

Surface treatment	Mechanico-thermal cycling	Alloy		Mean (SD)
		Gold	Co–Cr	
Tungsten bur	Absence	42.36 ± 6.45	40.18 ± 8.05	41.27 ± 7.18
APA 10 mm		48.75 ± 3.80	50.51 ± 9.71	49.63 ± 7.23
APA 20 mm		50.55 ± 5.49	49.71 ± 6.53	50.13 ± 5.88
Tungsten bur	Presence	32.54 ± 4.53	32.15 ± 5.68	32.34 ± 5.01
APA 10 mm		41.06 ± 8.19	43.12 ± 11.53	42.09 ± 9.79
APA 20 mm		40.58 ± 6.38	44.67 ± 12.52	42.62 ± 9.90
Mean (SD)		42.64 ± 8.25	43.39 ± 10.89	

Table 4 Results of three-way ANOVA for the data obtained by experimental conditions

Effect	DF	SS	MS	*F*	*p*
Alloy	1	16.80	16.80	0.27	0.602
Surface treatment	2	2316.14	1158.07	18.83	0.001*
Mechanico-thermal cycling	1	1915.04	1915.04	31.13	0.001*
Alloy/surface treatment	2	62.64	31.32	0.51	0.602
Alloy/mechanico-thermal cycling	1	41.02	41.02	0.67	0.416
Surface treatment/mechanico-thermal cycling	2	13.23	6.61	0.11	0.898
Alloy/surface treatment/ mechanico-thermal cycling	2	27.97	13.98	0.23	0.797
Residue	108	6643.36	61.51		
Total	119	11,036.20			

*Statistically significant difference at the level of 5%; R^2 (adj) = 33.67%.

DF, degrees of freedom; SS, sum of squares; MS, mean ratio square; *F*, probability; *p*, *p*-value.

metal and ceramic for three of the five tested Co–Cr alloys was similar to the shear bond strength values found for the ceramic and gold alloy. Wood et al[13] compared the flexural bond strength of two ceramics and two metallic alloys (base and gold) and also observed that the type of metallic alloy did not produce a significant effect.

Several in vitro tests have been proposed measuring the bond strength between the metal and the ceramic, including traction tests,[25] shear bond strength,[1,9,20] three-point flexural bond strength,[3,17–19] and four-point flexural bond strength,[16] since there is no consensus in the literature regarding the best test to evaluate bond strength between these two materials; however, Della Bona and Van Noort,[26] by analyzing the shear bond strength test, observed that this type of test generates arc-shaped cohesive fractures on all specimens. This type of fracture occurs due to the highly non-uniform stress distribution on the interface between the materials. On the other hand, the flexural bond strength test most closely simulates clinical conditions, because the specimens simultaneously suffer shearing, traction, and compression forces during the test.[27] Additionally, the three-point flexural bond strength test is recommended by the International Organization for Standardization[22] to analyze metal/ceramic bond strength. Therefore, the three-point flexural bond strength test was chosen for this study. Regardless of the evaluated group, flexural strength mean values were higher than the 25 MPa minimum established by ISO 9693.[23]

Anusavice[28] and Craig[29] suggested that airborne-particle abrasion of a bonding surface increases the metal surface energy, improving the wettability of opaque ceramic and, consequently, the bond strength, through micromechanical bonding; however, excessive roughness on the metal surface can create little gaps, which favor bubble formation on the interface during ceramic firing, damaging the bond between metal and ceramic.[30] In the present study, three types of metallic surface treatment were used for both metallic

Table 5 Tukey (5%) test for flexural strength values (MPa) for surface treatment conditions

Surface treatment	Mean ± SD	{1} – 36.81	{2} – 45.86	{3} – 46.38
1 Tungsten bur	36.81 ± 7.60		*p* = 0.000117	*p* = 0.000115
2 APA 10 mm	45.86 ± 9.31			*p* = 0.953382
3 APA 20 mm	46.38 ± 8.89			

Table 6 Incidence of failure types (%) after flexural bond strength test

Experimental groups (n = 20)	Failure types	
	Adhesive	Mixed
Gold/tungsten bur	100	0
Gold/tungsten bur/APA 10 mm	0	100
Gold/tungsten bur/APA 20 mm	0	100
Co–Cr/tungsten bur	100	0
Co–Cr/tungsten bur/APA 10 mm	0	100
Co–Cr/tungsten bur/APA 20 mm	0	100

Adhesive—along the interfacial region between the opaque ceramic and the metal; mixed—mixture of adhesive failure between the opaque ceramic and the metal with cohesive fracture of the ceramic.

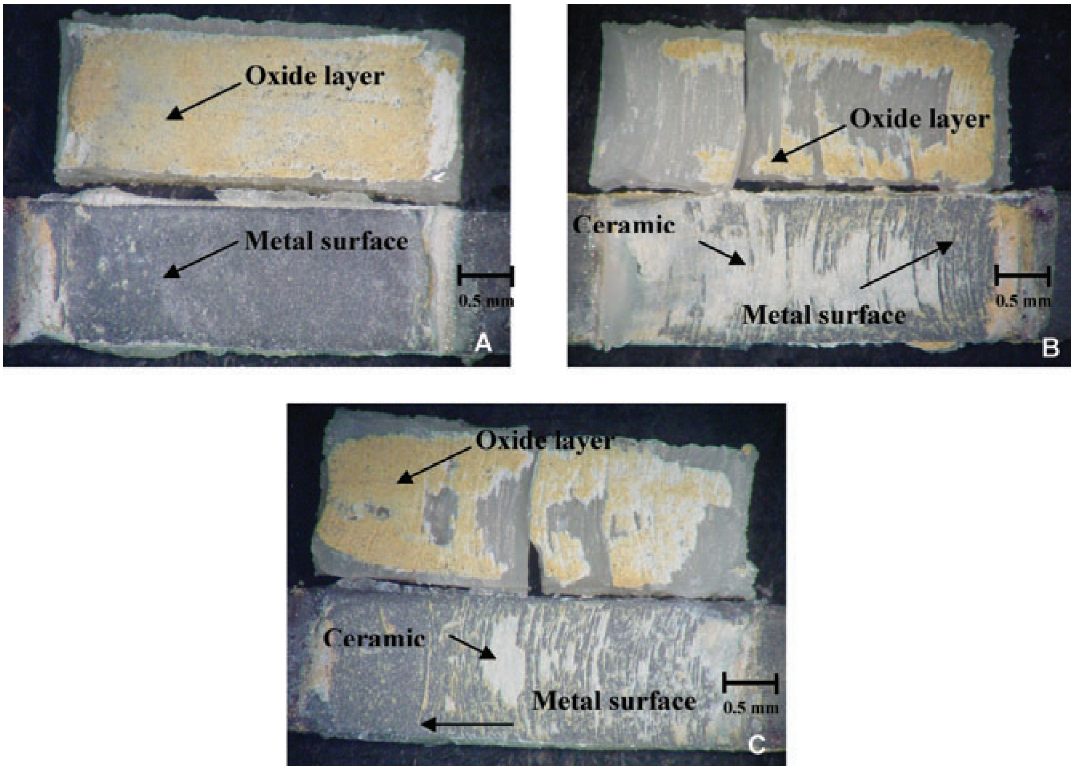

Figure 4 A–C Optical microscopic images of the Vita VM13/Au specimen (30×) after flexural strength test. (A) showing neither ceramic nor oxide layer remnants on the Au surface for Gr1, (B) opaque layer remnants on Au surface for groups Gr2, and (C) Gr3.

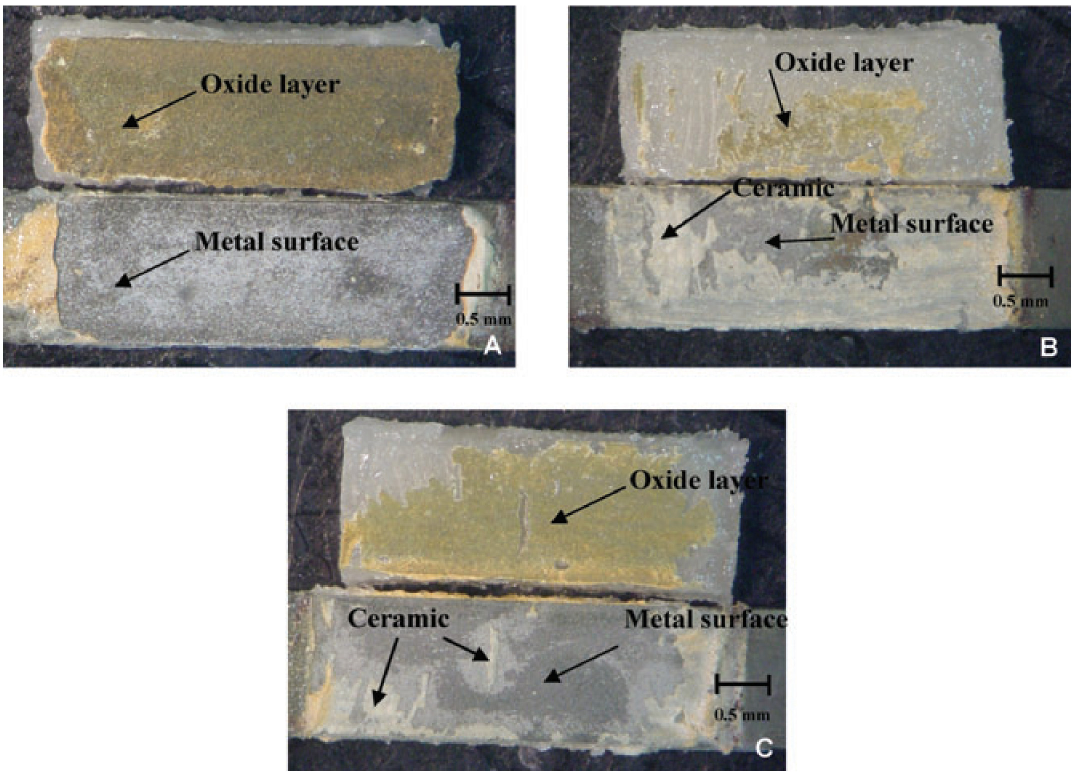

Figure 5 A-C Optical microscopic images of the Vita VM13/Co–Cr specimen (30×) after flexural strength test. (A) showing neither ceramic nor oxide layer remnants on the Co–Cr surface for Gr4, (B) opaque layer remnants on Co–Cr surface for groups Gr5, and (C) Gr6.

alloys before the application of ceramic. Air abrasion at APA 10 and 20 mm surface treatments, for both alloys, was quite similar in mean flexural strength values, and both had significantly higher bond strength than the tungsten bur treatment. Fischer et al[19] evaluated the effect of APA with Al_2O_3 on flexural strength between a feldspathic ceramic and noble alloys. They found that APA with Al_2O_3 significantly increased the metal/ceramic bond strength; however, this study compared the bond strength of 1 μm diamond paste metallographically polished metallic surface specimens with an airborne-particle-abraded metallic surface. Hofstede et al[17] studied the influence of metal surface finishing on the bond between a noble alloy and a ceramic, varying the use of APA with Al_2O_3 and the metal's finishing direction with aluminum oxide stone (unidirectional and bidirectional). Lombardo et al[20] compared the effect of metal surface treatment with tungsten burs and APA with Al_2O_3 on the bond strength between a basic alloy and two ceramics. Both studies concluded that APA with Al_2O_3 improved the bond strength values between metal and ceramic, confirming the results of the present study. According to Lombardo et al,[20] APA with Al_2O_3 created microretention on the metallic surface, contributing to the higher bond strength between metal and ceramic, whereas the tungsten burs produced macroretention less efficiently than the other surface treatment.

The highest recorded bond strength between metal and ceramic is shown when the fracture occurs inside the ceramic and not at the interface.[11,31] In the present study, after the specimens were evaluated by visual inspection, the presence of an opaque layer and ceramic was observed on the metal surface, and eruptions of the oxide layer were observed on the ceramic surface for the airborne particle abraded groups (Figs 4B, C and 5B, C). On the other hand, an oxide layer along the ceramic surface and the absence of opaque and ceramic layers over the metal surface were observed for the non-airborne-particle-abraded groups (Figs 4A and 5A). These results are in accordance with a study performed by Hofstede et al,[17] in which the highest bond strength between metal and ceramic occurred on specimens that received APA with Al_2O_3, observing mixed failures, while non-airborne-particle-abraded specimens presented adhesive failures at the metal/ceramic interface.

Most in vitro studies in dental research use static mechanical tests that do not represent the conditions of the aggressive mouth environment. The oral environment is capable of inducing physico-chemical changes on dental materials.[22] Induction of mechanical fatigue using computer models is a useful resource to estimate predictability of restorations to avoid catastrophic failures in vivo.[32] The exposure of metal ceramic restorations to temperature alterations during immersion in water induces repeated tensions that weaken the adhesion of materials due to the mismatch between the thermal expansion coefficient of the restoration components.[22] The results of this study showed that the presence of mechanico-thermal cycling did significantly interfere with the metal ceramic bond strength, that is, a decrease in the average bond strength of cycled groups was noted when compared to the non-cycled groups. This is in accordance with studies performed by Oyafuso et al[22] and Vasquez et al,[33] who used the same mechanico-thermal cycling conditions and observed a reduction in mean flexural strength values; however, this study indicated that the results of the groups without aging and with aging (mechanico-thermal cycling) were higher than established by ISO 9693[23] (25 MPa).

The results of this study showed that APA with Al_2O_3 at 10 and 20 mm improved the flexural bond strength between metals and ceramic used; however, one can speculate that the distance between the sandblaster nozzle and the specimen, larger than the distances used in this study, can decrease the efficiency of APA with Al_2O_3 and consequently decrease the bond strength between the ceramic and metal. Therefore, distance variations, as well as angulation, pressure, size of Al_2O_3 particles, and other alloys should be considered in future studies.

Conclusions

Based on the results and considering the levels adopted in this study, the following conclusions can be drawn:

(1) The average bond strengths were similar for the gold and Cr–Co alloys.
(2) APA with Al_2O_3 at 10 and 20 mm improved the flexural bond strength between the studied metals and ceramic.
(3) Mechanical cycling (20,000 cycles) and thermal cycling (3000 cycles) reduced the mean flexural strength values significantly for all six alloy/surface treatment combinations tested when compared to the control groups.

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