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Metal-Ceramic Bond Strength of Substrates Made With Different Fabrication Techniques: In Vitro Study

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ABSTRACT

The purpose of this in vitro study was to determine the metal-ceramic bond strength between dental porcelain and cobalt-chromium (Co-Cr) metal substrates fabricated by different techniques. Forty Co-Cr metal substrates were fabricated according to ISO 9693-1, by casting, milling, soft milling, and DMLS. Forty additional substrates were fabricated for each technique to record the modulus of elasticity. A commercially available feldspathic porcelain was placed on the substrates, and then the specimens were also tested for metal-ceramic bond strength with the 3-point bend test, according to ISO 9693-1. The fractured specimens were observed with optical and scanning electron microscopy using electron dispersive spectroscopy to define the mode of failure. X-ray diffraction spectroscopy was conducted to determine changes in crystalline phases after fabrication and the 3-point bend test. Statistical analysis was with 1-way analysis of variance and the Tukey post hoc test ($\alpha=0.05$). No statistically significant differences were found for modulus of elasticity among any of the groups. The metal-ceramic bond strength for casting had no statistically significant differences and the mode of failure in all groups was cohesive. The metallographic analysis of the as-received, the after porcelain firing, and the after 3-point bend test specimens revealed changes in microstructure. The crystallographic microstructure revealed that the patterns had minor changes among the groups. The study revealed that all of the techniques showed similar results. The modulus of elasticity and metal-ceramic bond strengths presented no statistically significant differences, and the mode of failure was cohesive.

Keywords. Metal-ceramic, Bond Strength, Casting, Milling, Soft Milling, DMLS, Laser Sintering.

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INTRODUCTION

Metal substrates for metal-ceramic restorations were traditionally constructed by casting dental alloys, but in recent years, new technologies have offered alternative solutions. The most common of these are the three-dimensional (3D) subtractive and additive techniques, such as milling and soft milling, which are methods that involve cutting the substrate from a soft material, followed by thermal treatment in an oven, and the direct metal laser sintering (DMLS) technique, which involves the construction of the metal substrates layer by layer with metal powder and a laser. Many researchers¹⁻⁹ have published articles concerning experiments with these techniques, using mostly cobalt-chromium (Co-Cr) alloys and testing different factors which influence the metal-ceramic bond strength. Also, many results have been published¹⁰⁻¹⁴ regarding bond strength values related to microstructural changes of alloys used under different processing and thermal conditions.

In addition, Co-Cr alloys have been utilized with these new technologies because they involve less concern about biocompatibility than the traditional cast nickel-chromium (Ni-Cr) alloys, for metal-ceramic prostheses. The purpose of the present research was to study the bond strength between Co-Cr alloys and a feldspathic dental porcelain when casting, milling, soft milling, and DMLS techniques were used, according to ISO 9693-1,¹⁵ to construct the substrates. The null hypothesis of the present study was that there would be no statistically significant differences in the metal and ceramic bond strengths for any of the fabrication techniques used for the construction of the metal substrates.

MATERIALS AND METHOD

Forty metal substrates were fabricated by casting, milling, soft milling, or DMLS techniques according to ISO 9693-1 requirements. The metal substrates were classified into 4 equal groups of 10, as shown in Table 1. In addition, 40 metal substrates were fabricated, 10 with each technique, to test the modulus of elasticity (E) (Table 1). Twenty-one plastic patterns were fabricated using custom-made equipment¹⁶ to the exact dimensions specified by ISO 9693-1 (length 25 ± 1 mm, width 3 ± 0.1 mm, and thickness 0.5 ± 0.05 mm).

Twenty of the specimens were positioned in casting rings, with an investment material (Giroinvest Super; Amann Girrbach) and then cast using a Co-Cr alloy (Phase-C3; Unitech). The investment material was removed by sandblasting with 110- μ m Al₂O₃ particles (Cobra; Renfert) with 200 kPa pressure. The one plastic pattern left was used for the scanning procedure (Ceramill Map 400; Amann Girrbach) to create the digital prototype, and a standard tessellation language (STL) file was created for the milling, soft milling, and DMLS techniques.

The milling was performed with a YenaDentD40 5-axis cutting machine (YenaDent Europe), using a Co-Cr 10-mm Magnum Splendidum 4 disc (Mesa).

The soft milling was performed with a Ceramill Motion 2 5-axis cutting machine (Amann Girrbach) using a 10-mm Co-Cr special soft disc Ceramill Sintron from the same company for the milling. After the soft milling procedure, all of the substrates of this group were subjected to thermal processing in a Ceramill Argotherm 2 furnace (Amann Girrbach) to acquire the optimum mechanical properties.

The DMLS substrates were created using the 3Shape program with the STL file, and then with an MLab curing machine (Concept Laser) using the 10-30 μm Co-Cr powder Remanium Star CL (Dentaurum). The compositions of the manufacturers' Co-Cr alloys are presented in Table 2.

Ten substrates from each group were submitted to a 3-point bend test in a universal testing machine (Tensometer10; Monsanto). A standard load was applied with a crosshead speed of 1.5 mm/min and a distance between the supporting points of 20 mm. The E was calculated using the following formula: $E = \frac{L^3 \Delta P}{4bh^3 \Delta d}$, where L is the distance between the supporting rods (20 mm), b is the width of the specimen (3 mm), h is the thickness of the specimen (0.5 mm), ΔP and Δd are the load and deflection increment, respectively, between the 2 specific points in the elastic portion of the curves.

The porcelain was applied in layers. The first layer was the bonding agent (Metabond; Unitech), the second and third layers were the opaque and dentin of Noritake EX-3 dental feldspathic porcelain (Kuraray; Noritake Dental). The ceramic materials were applied centrically on 1 side of the metal substrate using custom-made equipment adjusted to the dimensions specified in the ISO 9693-1 requirements (length 8 ± 0.1 mm, width 3 ± 0.1 mm, thickness 1.1 ± 0.1 mm). The specimens were fired at $980 \pm 5^\circ\text{C}$ with the temperature increasing at rate of $80^\circ\text{C}/\text{min}$ in vacuum conditions in a Dekema Dental Keramik oven (Austromat 624). After the preparation of the metal-ceramic specimens, a 3-point bend test was performed with the same equipment (Tensometer10; Monsanto) that was used for the definition of E. The load was applied on the opposite side from the porcelain layers. Fracture diagrams were obtained, and the bonding strength was calculated using the following formula: $\sigma = \frac{3FL}{2bd^2}$ where σ is the tension, F is the largest amount of weight that is applied (N), L is the distance between the 2 supporting rods (mm), b is the width of the specimen (mm), and d is the thickness (mm). An optical stereoscopic microscope (Eclipse ME 600; Nikon-Kogaku) was used to define the mode of failure of the fractured parts of the specimens of each group (Fig. 1) under reflected light, at $\times 5$ magnification. To verify the result of the present study, these areas were recorded to measure the percentage of the ceramic mass, for the next measurements regarding the modes of failure.

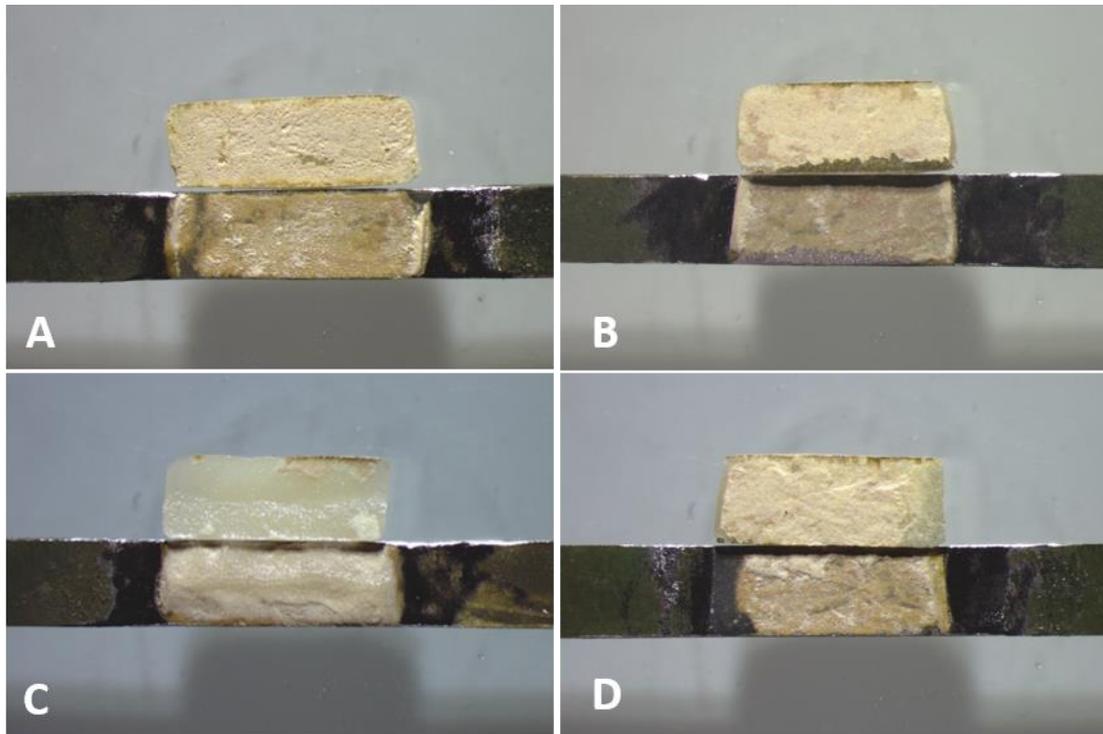


Figure 1: Metal-ceramic specimens after 3-point bend test (original magnification $\times 5$). A, Cast. B, Milled. C, Soft milled. D, direct metal laser sintered.

To verify the kind of material of the different colored areas recorded with optical microscopy, some selected areas of the fractured surfaces were observed in a scanning electron microscope (SEM; JEOL 6380LV) operating at an accelerating voltage of 30 kV, by secondary electron images at $\times 100$ magnification. The qualitative and quantitative definitions of the elemental distribution were obtained by X-ray energy dispersive spectroscopy (EDS) using a super ultrathin beryllium window (Sapphire; Edax Intl). The EDS area (mapping) analysis was obtained in representative metal substrates as received from the manufacturer, for each group, as well as the bonding agent and opaque porcelain before their use on the substrates. The EDS analysis was also conducted on specific areas of the fractured specimens.

Cohesive failure was defined as $>50\%$ of the fractured surface of the specimen covered by ceramic material (including bonding agent), whereas adhesive failure was defined as $<50\%$. The mode of failure in all of the groups in this study was cohesive.

Phase composition was analyzed by X-ray diffraction (XRD) using a Philips X'Pert diffractometer operating with $\text{CuK}\alpha=1.54056 \text{ \AA}$, scanning speed of $0.01^\circ/\text{s}$ from 10° to 90° , and scanning range of 2θ , with metallographic preparation of the patterns to observe the grain size by microscopy. The peaks for the 4 techniques were identified by comparing every alloy processed, with standard files from International Centre for Diffraction Data (ICDD; documents 96-901-2928 for Co-fcc, 96-901-1618 for Co-hcp, and 96-231-0285 for intermetallic Co_7Cr_8). Additives were also identified by 01-076-0319 for TiO_2 and 00-004-0593 for CeO_2 . To compare the processing method for every Co-Cr alloy of the 4 groups used

in the present study, XRD patterns were used and divided into groups of as-received, after porcelain firing, and after 3-point bend test patterns. The mean value and standard deviation were calculated for E.

Bond strength was statistically analyzed by 1-way analysis of variance (ANOVA) and Tukey post hoc test; $P < .05$ was considered statistically significant.

RESULTS AND DISCUSSION

The results for E of the 4 different techniques applied for the fabrication of the metal substrates are presented in Table 3. No statistically significant difference was recorded among the tested groups ($P = 0.268$). The results for metal-ceramic bond strength are presented in Table 4. Again, no statistically significant difference was recorded among the tested groups ($P < 0.173$). The results of the EDS area analysis of the as-received ceramic materials (bonding agent, porcelain) revealed that the bonding agent presented an increased amount of Ti, while Zr and Si were the main elements detected in the opaque porcelain. The mode of failure for all groups was cohesive.

Also, XRD analysis of the used alloys was conducted. The 2 typical primary phases of the casting group of Co-Cr alloys, γ -fcc (γ -Co-Cr-fcc) and ϵ -hcp (ϵ -Co-Cr-hcp), were found in the as-received alloys. Also another phase that fit with intermetallic Co_7Cr_8 was revealed. After porcelain firing and after the 3-point bend test, the Co-Cr-fcc became the major phase. In addition, TiO_2 and CeO_2 were identified showing that the presence of porcelain and of Co-Cr-hcp could not be discounted, because their peaks appeared in the same place as those of the additives from opaque porcelain. The amorphous halo observed at low angles (below 25 degrees) in the after porcelain firing group and after the 3-point bend test, was the result of their additive content. As in the casting specimens, the patterns of the as-received milling group showed the 2 fcc/hcp Co-Cr phases together with an intermetallic Co_7Cr_8 phase. In APF-A3PBT patterns, the Co-Cr-fcc became the major phase, in combination with the TiO_2 and CeO_2 phases. The amount of intermetallic phase decreased. The results recorded for the Co-Cr alloys from the soft milling technique revealed that the major phase was Co-Cr-hcp, whereas the Co-Cr-fcc phase was practically undetectable. Intermetallic Co_7Cr_8 peaks were also identified. In the patterns for the after porcelain firing group and the 3-point bend test, only the presence of Co-Cr-hcp phase was recorded, while no intermetallic peaks were detected. The DMLS patterns for as-received specimens showed the same phases as the casting and milling specimens. In the patterns for after porcelain firing and the 3-point bend test, the intermetallic peaks disappear, and the presence of Co-Cr-fcc phase becomes much more evident. The components TiO_2 and CeO_2 were clearly detected, as in the patterns referred to previously in the after porcelain firing group and the 3-point bend test.

Table 1: Study groups

Groups	Substrates for Testing Metal-Ceramic Bond Strength (n)	Substrates for Testing Modulus of Elasticity of Alloys (n)
Cast	10	10
Milled	10	10
Soft milled	10	10
DMLS*	10	10

*DMLS, direct metal laser sintered.

Table 2: Compositions of the alloys used provided by the manufacturers

Co-Cr Alloys and Manufacturers	Composition (wt %)	Lot/Patch Number	Country of Origin
Phase-C3, Unitech	Cr, 28%; W, 8.5%; Si, 1.5%; Nb, 1%; Co, 60.5%; C, 0.02%	PHC74	France
Magnum Splendidum Type 4, Mesa	Cr, 28%; W, 9%; Si, 1.5%; Nb, 1%; Co, 60%; Mn, Fe	10212 10	Italy
Ceramill Sintron, Amann Girschbach	Cr, 28%; Mo, 5%; Co, 66%; Mn, Si, Fe	1602011- 160	Austria
Remanium Star CL, Dentaurum	Cr, 28%; W, 9%; Si, 1.5%; Co, 60.5%; Mn, N, Nb, Fe	463369A	Germany

Table 3: Modulus of elasticity for the alloys tested^a

Groups	E (GPa)
Cast	229.8 ±25.4
Milled	216.8 ±21.9
Soft milled	251.8 ±19.9
DMLS*	228.3 ±20.1

*DMLS, direct metal laser sintered; E. modulus of elasticity. ^aValues are means ± standard deviation (n=10).

Table 4. Metal-ceramic bond strengths^a

Groups	Bond Strength (MPa)
Cast	44.8 ±7.9
Milled	48.4 ±4.5
Soft Milled	44.1 ±9.4
DMLS*	44.5 ±8.7

*DMLS, direct metal laser sintered. ^aValues are means ± standard deviation (n=10).

DISCUSSION

According to the results of the present study, the null hypothesis was verified, and no statistically significant difference was found among the tested groups concerning the metal-ceramic bond strength. The results for modulus of elasticity of all tested Co-Cr alloys were in accordance with the data provided by the companies and that reported in the literature. Regarding XRD, the pure solid cobalt was (under equilibrium conditions) face-centered cubic (fcc) above 419°C and hexagonal close-packed (hcp) below 419°C.¹⁷ The pure solid Cr showed the body-centered cubic phase (Cr-bcc) as departure phase; however, the solid-state transformation of Co from fcc to hcp was slow, so the C-fcc phase was retained under normal

conditions. Changes which were related to fcc or hcp Co-Cr could be explained by the use of mechanical or thermal treatments based on the departure phases. Reference works on dental materials¹⁸ refer to E values for Co-Cr alloy in MPa, and Dimitriadis *et al*⁴ found E values of 222 and 227 MPa for Co-Cr alloys used in casting and DMLS methods, respectively. Most researchers^{1,2,4-9} who have studied metal-ceramic bond strength *in vitro* using casting and selective laser melting (SLM) techniques based on ISO 9693 have found fracture values between 37 and 47 MPa for casting and 45 and 50 MPa for DMLS, with no statistically significant differences. These results are consistent with the results of the present study (44.8-44.5 MPa), where also no statistically significant differences were recorded. Exceptions to these fracture values, including the high values recorded by Lawaf *et al*³ between cast (74.94 ±16.06 MPa) and SLM (69.02 ±5.77 MPa) groups, and low values recorded by Li *et al*¹ (cast 32.15 ±2.39 MPa and SLM 32.31 ±3.06 MPa), can attributed to the different procedures followed during the fabrication of the specimens.

Researchers who have studied metal-ceramic bond strength for metal substrates produced by milling^{1,2} have found fracture values between 34 and 43.3 MPa, which are slightly less than the results of the present study (48.4 MPa). Other researchers followed different fracture testing procedures to record metal-ceramic bond strength using ANSI/ADA No. 388 and shear tests.^{9,19} They found no significant difference in bond strength between cast and selective laser sintering (SLS) techniques. In particular, Lee *et al*¹⁹ studied metal-ceramic shear bond strength in specimens produced by soft milling, and found a value of 35.1 MPa, which was close to the value recorded in the present study (44.1 MPa).

Most of the previously mentioned studies were accompanied by testing for mode of failure. The results revealed cohesive fractures in the majority of the tests, independent of the fracture method used.¹⁻⁵ Many researchers report mixed adhesive and cohesive failure, with cohesive failure the more prevalent.^{4,5,9,19}

Al Jabbari *et al*¹⁰ compared the metallurgical microstructures of Co-Cr dental alloys fabricated via casting, milling, and SLM. Different microstructures were identified among the groups. Their study also focused on the interfacial characterization of Co-Cr porcelain fused to metal (PFM) alloys fabricated using casting, milling, and SLS. Kim *et al*¹² evaluated the microstructure and mechanical properties of Co-Cr dental alloys fabricated by 3 computer-aided design and computer-aided manufacturing (CAD-CAM) techniques and compared them with the traditional casting technique. They found that the microstructure of the alloys was strongly influenced by the manufacturing processes, and the SLM group showed superior mechanical properties. Several studies have been conducted regarding the DMLS technique: Wu *et al*⁷ studied the microstructure of Co-Cr dental alloy using the SLM technique for the specimens, and compared it with casting. The microstructure of the SLM group appeared to be

oriented, which gives good mechanical properties. The results of elemental analysis using SEM/EDS revealed that SLM showed slightly better bond strength due to the intermediate layer between the metal substrate and the porcelain. Barucca et al¹¹ used Co-Cr-Mo specimens produced by DMLS for hardness and structural characterization compared with cast ones and analyzed by XRD, SEM, transmission electron microscopy, and energy-dispersive X-ray spectroscopy, revealing that the face-centered cubic phase of the sintered alloy showed increased hardness.

A study by Koutsoukis et al¹³ showed the advantages of using the SLM technique for substrate fabrication in dental structures, in comparison with casting and milling techniques. Li et al,¹⁴ in an experimental study, produced Co-Cr specimens by casting, powder metallurgy, and CAD-CAM, and analyzed those using EBSD EDS and XRD. Specimens were treated using different porcelain firings, and crystallography, grain size, and chemical composition were analyzed. Krug et al¹⁸ did a comparison of 3 different forces on metal-ceramic systems fabricated using 3 different techniques. Specimens were subjected to thermal and mechanical aging. The only statistically significant difference was recorded in force maximum for specimens without thermal and mechanical aging. Thermal versus mechanical aging did not significantly affect the result. Serra-Prat et al²⁰ conducted shear bond strength tests to determine the metal-ceramic bond strengths for milling, casting, and laser sintering (LS) techniques, after thermocycling procedure, and analyzed the results using 2-way ANOVA. They found that there were no statistically significant differences among the 3 techniques, between either the thermocycled or nonthermocycled specimens of the LS group, in contrast to the cast and milled groups where they observed adhesion differences. Suleiman et al²¹ made identical crowns with milled, cast, and LS alloy using a Co-Cr and high gold alloy. There were no statistically significant differences among the 3 groups for fracture strength, whereas high gold alloy crowns revealed statistically significantly higher metal-ceramic bond strength. Tuna et al²² conducted an electrochemical corrosion experiment for metal substrates produced by milling, casting, or LS using a Co-Cr alloy, and found that the corrosion resistance was greater in the LS group than in the cast group. Kruth et al²³ compared the mechanisms of the SLS and SLM techniques, which can be classified into the categories of solid state sintering, chemically induced binding, and liquid phase sintering. In this paper, they describe all of the possible results that can be achieved by these techniques, including metal constructions as well. Also, as it can be observed in tables 3 and 4, the sources of variation given for the individual specimen measurements for the elastic modulus and metal-ceramic bond strength are close enough to the results given at the similar studies.

This present study had some limitations, including the poor resolution of the SEM images and XRD graphics that were therefore difficult to provide to accompany the text. As a suggestion

for future research, it would be useful to analyze the longevity of each of these constructions, comparing the groups. Also, another possible study could include these 4 groups to reveal the best effect on tissues, and which one would be more suitable. Furthermore, it would be interesting to analyze the ions released from these materials. Given all of the new technologies that are now a part of the production of fixed dental prostheses and looking forward to new technologies that may emerge, a similar study including also 3D printers might give valuable insights.

CONCLUSION

Within the limitations of the present laboratory study, the following conclusions can be drawn: The modulus of elasticity of Co-Cr dental alloys fabricated by casting, milling, soft milling, or DMLS is similar for all fabrication methods. Metal-ceramic bond strength is satisfactory in all cases, covering the minimum acceptable value of the 25 MPa suggested by ISO 9693-1. The mode of failure can be expected to be cohesive, independent of the fabrication method of the metallic substrate. XRD analysis reveals differences in microstructure between the as-received metallic substrates and the fractured specimens within each group. Co-Cr-fcc is the major phase in the as-received alloys for casting, milling, and DMLS techniques. An increase in the formation of Co-Cr-fcc phase is observed after the porcelain firing cycle and the 3-point bend test for the casting, milling, and DMLS methods. The Co-Cr-fcc structure is not maintained with the soft milling technique, while Co-hcp is the dominant phase in the soft milling alloy before and after porcelain firing and the 3-point bend test. An intermetallic compound, Co_7Cr_8 , is formed in all of the as-received alloys. The presence of this intermetallic compound drastically decreases after porcelain firing and the 3-point bend test for cast and milled alloys, and completely disappears with the soft milling and DMLS techniques.

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