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A comparative evaluation of bond strength of ceramic to metal alloys fabricated through conventional lost-wax technique and direct metal laser sintering: An in-vitro study.

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Abstract

Purpose**:** The purpose of this study was to compare and evaluate the bond strength of ceramic to different metal alloys i.e. cobalt-chromium (Co-Cr) and nickel-chromium-titanium (Ni-Cr-Ti), fabricated through conventional lost-wax technique and direct metal laser sintering (DMLS).

Methods**:** Sixty-three metal plates (n=21 per group) of 25x3x0.5mm were prepared with conventional lost wax (Co-Cr and TiLite) and DMLS (Co-Cr) technique; and ceramic was layered at the center. Group I (cast Co-Cr), group II (DMLS Co-Cr) and group III (cast TiLite) were compared. A three-point bend test was applied using a universal testing machine to measure the flexural bond strength. Fractured specimens were observed to classify the type of failure. The statistical analysis was carried out using descriptive analysis (mean, SD, CI) and Tukey Honest Significant differences test.

Results**:** The means for flexural strength for Group I, II and III were found to be (20.24N ± 4.38), (15.95N ± 3.52), and (12.60N ± 2.36) respectively. Comparison between individual groups were made with Tukey's Honest Significant Difference *post-hoc* test and it was found that Group I had higher bond strength than Group II and Group III. Most of the metal–ceramic specimens in the 3 groups exhibited a cohesive failure.

Conclusion: It was concluded that, although statistically significant differences were found, the bending strength values of all groups were within the acceptable levels of 5N as in German Standard Institution DIN 13927. Cohesive bond failure percentage was more in all the three groups, suggesting an acceptable bond between metal and ceramic.

Keywords: metal-ceramic bond strength, flexural strength, three-point bend test, lost-wax technique, DMLS technique, Co-Cr, TiLite.

Introduction

Since decades, combination of base metal alloys and porcelain have been widely used for fabrication of fixed partial dentures or single crowns due to its good performance and esthetics[1] with excellent mechanical properties in low cost.[2] The compatibility of metal and ceramic is an important factor for metal-ceramic restorations[3,4] as its clinical success primarily depends on the bond strength between the metal substructure and ceramic.[5] With the availability of various alloys and the processing techniques, the clinicians face a baffling set of options.

Base metal alloys, such as Nickel-Chromium (Ni–Cr) and Cobalt-Chromium (Co–Cr) alloys have been in extensive use because of their mechanical properties, low cost[6,7] and the ability to allow the fabrication of thinner substructures with greater rigidity.[8] The modulus of elasticity of the Co-Cr alloys is the highest of any alloy systems.[9] Lately in 1700s, Titanium (Ti) was discovered, the excellent corrosion resistance showing a strong passivity trend and biocompatibility of its alloys made them attractive for hypersensitive patients especially.[10-12]

Apart from the material selection even the technique of fabrication plays an important role in metal-ceramic bonding. The conventional lost-wax technique is widely used for fabricating the metal copings. However, this is technique sensitive[13] because of alloy's high melting range and predisposition to form unstable oxides that cause bond failure at the interface. Also it is time-consuming and labor-intensive.[14] Thus, to overcome the labor errors, the direct metal laser sintering (DMLS) system was later developed which is an additive technology. It works on information received from the computer-aided design and using this data file, metal powder is shot selectively and fused with a laser to laminate approximately a 20–60µm thick layer with each shooting.[13] Unlike subtractive milling methods, it is less time consuming, minimize material consumption, and decrease labor costs. Moreover, they are capable of reproducing every detail in the CAD data, including undercuts and complex internal geometries.[2]

If these techniques or metal alloys are improperly selected, bonding failure can occur in metal-ceramic prosthesis[15], requiring repair or replacement of the prosthesis.[16,17] Thus, its clinical survival is directly related to a satisfactory bond between the metal and ceramic layers. For measuring this, different test designs have been used, including shear tests, tensile tests, a combination of shear and tension tests, bend tests, and torsion tests.[18] However, most of these tests show stress concentration effects instead of the stress distribution, and this may alter the results. Currently, the International Organization for Standardization (ISO) standard recommends the use of flexural strength testing machines with 3-point bending for the evaluation of metal-ceramic bond strength.[19]

As the metal-ceramic prosthesis continue to be used and because new fabrication technologies are being developed, the purpose of this in-vitro study was to compare and evaluate the metal-ceramic bond strength, with different metal alloys prepared by using the conventional method and DMLS method.

Materials and methods

A total of 63 samples (n=21 per group) of this design [Figure-1] were fabricated for the test. Each specimen was layered with ceramic at the centre. The three groups were:

Group-I: Metal substructure fabricated through conventional lost wax technique using Cobalt-Chromium base metal alloy (Cast-CoCr)

Group-II: Metal substructure fabricated through DMLS using Cobalt-Chromium base metal alloy (DMLS-CoCr)

Group-III: Metal substructure fabricated through conventional lost wax technique using Nickel-Chromium-Titanium base metal alloy (Cast-TiLite)

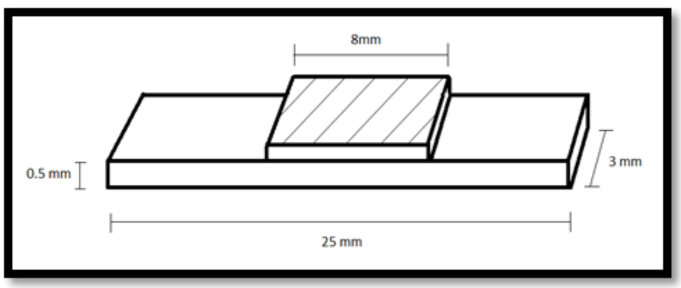

Figure 1: Schematic diagram of the specimen

The composition of each alloy is found in weight percent is as mentioned in Table-1.

Table-1: Composition of alloys used for each group

Group	Alloy	Composition
I	Cast-CoCr (Wirobond)	63.3 (Co), 24.8(Cr), 5.3(W), 5.1(Mo), 1.0(Si), <1.0(Ce)
II	DMLS-CoCr (EOS CobaltChrome SP2)	63.8(Co), 24.7(Cr), 5.4(W), 5.1(Mo), 1.0(Si), 0.5(Fe), 0.5(Mn)
III	Cast-TiLite (TiLite Premium alloy)	75(Ni), 13(Cr), 5(Ti), 5(Mo), <0.1(Co), 2(Al)

Fabrication of metal plates

To make the cast alloy specimens i.e. group I and III, acrylic resin plates were fabricated with dimensions of 25×3×0.5 mm, according to ISO9693 standard.[19] A putty (Express™ XT Putty Soft, 3M ESPE) impression of this plate was made to prepare a mould to fabricate the wax patterns (Crown Wax, BEGO). Twenty-one wax patterns were fabricated for each of Co-Cr (Wirobond) and Ni-Cr-Ti (TiLite) and invested with phosphate-bonded investment powder. Then casting for Co-Cr and Ni-Cr-Ti alloy was carried out on an induction casting machine (Unident Manual Centrifuge, India). All casting rings were bench-cooled, divested and sand blasted to retrieved the casting.

For specimens produced by direct metal laser sintering (DMLS), design was created in CAD software (Exocad) and converted to standard tessellation language(stl) files, which were transmitted to the DMLS equipment (Eplus EP 150M, Shining3D). The laser sintering system had a radiation heater, a focused laser beam at the roof, a platform on the floor with metal powder on both the sides of the platform, which is adjusted by a movable piston. The metal powder (EOS CobaltChrome SP2) of very fine particles (20 microns), was spread evenly on the platform and the laser beam with 200W power melted the metal locally to fuse the powder to form the metal plate.

All the plates were finished using metal finishing points (Metal finishing and polishing kit – Shofu Inc.) in the same way to achieve the desired dimensions. Then, they were cleaned in distilled water for 2-3 min to remove all the metal particles, other debris and oil which can affect the wetting surface. The dimensions of all specimens were confirmed with a 0.02-mm precision digital calliper (Aerospace). The specimens were then airborne-particle abraded for 10 seconds at a pressure of 0.2 MPa, using 150-microns Al_2O_3 particles at an angle of 45° from a distance of approximately 1 cm. The metal specimens were then oxidized in a ceramic furnace (Dentsply Multimat NTX Press) at 950°C without vacuum for 1 min as recommended by the manufacturer. The surfaces of all specimens exhibited a grey appearance after oxidation firing

Ceramic layering

A conventional multi-layer leucite containing veneering ceramic(Ivoclar Vivadent IPS Inline ceramic system, Ivoclar Vivadent AG, Liechtenstein) was used for layering. Two layers of opaque ceramic of up to 0.1 mm thickness each were applied at the centre of one side of each metal plate using standard ceramic build-up approach. The rectangular area of applied ceramic had dimensions of 8 x 3 mm. The furnace was programmed as recommended by the manufacturer for firing opaque ceramic, which required 25 minutes. The final opaque ceramic thickness was approximately 0.1-0.2 mm. The first layer of body ceramic (0.5 mm) was fired for 25 min and the second layer (0.3 mm) for 17 min. Thus, the final thickness of ceramic layers was 0.8±0.1 mm, which was measured using metal gauge. A small amount of glaze liquid was rubbed on the ceramic area, and self-glazing at 500ºC for 16 min was performed using the glaze cycle.

Flexural strength test

All the 63 specimens were subjected to a three-point bending test as recommended by ANSI/ADA Specification No.38 and current ISO specification 9693 for measuring the metal-ceramic bond strength. This test was conducted with a universal testing machine (Praj metallurgical laboratory, Kothrud, Pune). Figure-2 shows the bending apparatus that was used for bond testing (UNITEST-10; ACME Engineers, Pune). The distance between the bottom supports (support span) was kept 20mm and the pending piston radius was 1 mm. All the specimens were positioned such that the ceramic layer was on the opposite side to the applied load. A constant rate of displacement (cross-head speed) of the loading member of 1.5 mm/min was employed, and the force was recorded when fracture or separation of ceramic layer occurred [Figure-3] (up to crack initiation). The load that led to the initial separation of materials (debonding strength), for each specimen was obtained in newton(N).

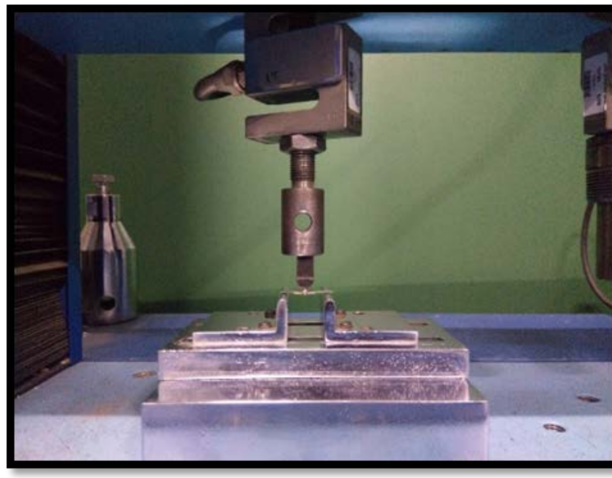

Figure 2: Universal testing machine

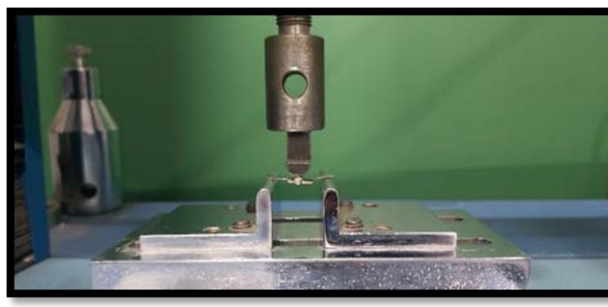

Figure 3: Three-point bend test

Figure 4: Adhesive failure

Figure-5: Cohesive failure

Observation of the type of bond failure

All fractured specimens were observed with the naked eye, and representative images were made using a DSLR camera. The types of bond failure were classified as adhesive, cohesive and mixed. The failure mode was defined to be adhesive when failure was between the metal and the ceramic and more than 75% metal substructure was visible [Figure-4], cohesive when failure was entirely within the ceramic or more than 75% of metal substructure was covered with ceramic or opaque layer [Figure-5], and all the other cases were said to be mixed mode or showing a combination of adhesive and cohesive failure [Figure-6].

Figure-6: Mixed failure

Statistical analysis

Data obtained was evaluated using descriptive analysis for mean, standard deviation (SD), confidence interval (CI) and Tukey Honest Significant differences test was applied for comparison between the groups.

Results

The data was analysed using SPSS20 software. The obtained value set was checked for its distribution through normal probability plot. The quantitative data was subjected to descriptive analysis for mean ± SD. The results were analysed with Tukey Honest Significance test for intergroup analysis.

Flexural bond strength values

The mean values and SD for the metal-ceramic bond strengths were obtained in Newtons(N). The highest mean flexural bond strength (20.24N) was observed in group-I (Cast CoCr), whereas the lowest mean flexural bond strength (12.60N) in group-III (Cast TiLite). Table-2 shows the highest mean flexural bond strength, SD, along with the highest and lowest values for the specimens.

Table-2: Flexural bond strength values of groups

Groups	N	Mean(N)	SD	Min(N)	Max(N)
I	21	20.24	4.38	14.5	34
II	21	15.95	3.52	10	24
III	21	12.60	2.36	8	17.5

Inter-group Analysis

According to test results a statistically significant difference ($p < .05$) existed between all the groups with 5% level of significance. It was also seen that the mean difference is also statistically significant and is highest among group I and group III ($p < .001$). Table-3 depicts the statistical differences between the groups.

Table 3: Tukey Honest Significant Differences Test Results

(I) Groups	(J) Groups	Mean Difference (I-J)	Std. Error	P value (Significance)	95% Confidence Interval	
					Lower Bound	Upper Bound
I	II	4.28571*	1.08604	.001	1.6757	6.8957
	III	7.64286*	1.08604	.000	5.0329	10.2528
II	I	-4.28571*	1.08604	.001	-6.8957	-1.6757
	III	3.35714*	1.08604	.008	.7472	5.9671
III	I	-7.64286*	1.08604	.000	-10.2528	-5.0329
	II	-3.35714*	1.08604	.008	-5.9671	-.7472

Type of Failure Count

The types of failures (adhesive, cohesive and mixed) observed are given in Table-4. It was noted that highest share of cohesive failure was seen in all the groups.

Table 4: Types of Failures

			Adhesive	Cohesive	Mixed
Groups	I	Count	3	14	4
		percentage	14.3%	66.7%	19.1%
	II	Count	3	16	2
		percentage	14.3%	76.2%	9.5%
	III	Count	1	13	7
		percentage	4.8%	61.9%	33.3%

Discussion

Metal-ceramic prosthesis are used in dentistry for 30-40 years.[20] Over the past several decades, different tests have been used to evaluate the bond strength of dental materials.[21] Lenz et al[22] pointed out that the bonding between ceramic and alloy is complicated because many variables interfere during the fabrication. Studies have shown that there is no ideal test that can measure the shear forces at the metal-ceramic interface.[23-25] However, in the current ISO specification 9693[19] employs a three-point bending test because it better simulates clinical conditions; as the specimens are under compression, traction, and shear bond strength simultaneously.[26]

Clinically, fracture may occur especially when a new material or technique has been used.[24] As it is known that metal-ceramic bond depends on the chemical interaction between the ceramic and the metal alloy, thus their appropriate selection is important. Most widely used alloys are Ni-Cr and Co-Cr base metal alloys.[27] Thus, the alloys chosen were aimed to represent popular choices made. And titanium (Ti) alloy, as it has been investigated in dentistry for its benefits. Apart from alloy selection, various techniques are available for fabrication of metal substructure. DMLS having advantage of fabrication of restorations with a uniform quality in lower cost was used to compare with the conventional technique.

In our study, the casted Co-Cr group showed higher flexural strength compared to the DMLS-Co-Cr and Cast-TiLite group. The results are in accordance with that of Akova et al[12] and Dimitriadis et al[28], which showed higher bond strength of casted group compared to the DMLS group; and Singh A. et al[29] that showed higher bond strength of casted Cobalt-Chromium over Casted-TiLite group, though the difference was not statistically significant. One reason for higher bond strength of the casted group can be the micro-mechanical interlocking of ceramic to metal due to its surface morphology. The cast specimen mainly consists of dendritic-like morphology, leading to penetration of ceramic powder. It had an austenitic matrix and carbide content enriched along the grain boundaries. While, the DMLS alloy showed a dense austenitic matrix with fine grains, without an obvious precipitated phase. [2,26,30] The skin region of each powder particle is completely melted with rapid solidification resulting in compact structures of upto 100% density with small grain size[31]. So, in the case of densely sintered ceramics, the higher density and lower porosity of sintered materials make them resistant to chemo-mechanical treatments.[32,33]

Surface roughness is another parameter which aids in micro-mechanical interlocking. Generally, it is considered that higher roughness would lead to increased bond strength due to improved contact area[34,35] and increase wettability.[5,36,37] It is affected by the airborne abrasion particle size. Studies[38-40] suggest that more than 50 mm to upto 200mm particle size showed improved metal-ceramic bond, hence a 150 mm particle size was used for all groups in our study to eliminate bias. Akova et al[12] had also used the same size and the results found were similar to our study.

Bae et al[41] considered that the bond strength of DMLS Cobalt-Chromium alloys could be improved by the lamellar morphology. In their study, after air abrasion with 50-mm particles, the DMLS Cobalt-Chromium alloy surface exhibited 100-mm-thick layers aligned with a gap between them. They believed that the bond strength increased because of the ceramic powder penetrating these gaps. The DMLS specimens in our study may not have such laminated structure, because the lamination thickness in the laser irradiation was set at a low thickness of 25 mm, whereas air abrasion was done with 150 mm particle size, allowing the particles to remove the laminated structure. The relatively higher roughness of the DMLS group could be related to the adherence of partially melted powder particles on the alloy surface. Fox et al.[42] studied the effect of process parameters on the surface roughness and revealed a large number of the partially melted powder particles on the surfaces, that lead to partial fusion of isolated powder particles during fabrication, which might affect the roughness (balling phenomenon).[41] Thus, extensive roughness may reduce the bond between alloy and ceramic, and this might explain why the DMLS group did not represent the strongest bond. However, the surface roughness or alloy morphology of DMLS substrates is also influenced by the manufacturing parameters, like the powder particle size, material composition, layer thickness, geometry of the object, building direction, scan speed and laser specifications, further affecting the bond strength.[41,43]

Many researches[2,12,28,30,41,44-46] studied the metal-ceramic bond strength when the DMLS technique was used. Some of them[12,44] tested applying the shear test whereas others[2,28,30,41,45,46] used the three-point bend test. All researches presented no statistically significant difference of the metal-ceramic bond strength independently of the test used. Absolute differences which were recorded among the previously mentioned studies waved between 70 MPa to 31 MPa, can be attributed to the different Cobalt-Chromium dental alloys and feldspathic porcelains used. Also, superficial roughness of the metal substrates due to different porosity between the two applied techniques may affect the overall bond strength.

The significant difference between the casted TiLite group and the other two groups in our study may be attributed to careful maintenance of firing temperature and reactions with oxygen during casting. For PFM restorations on Ti castings, the ceramic fusion temperature must be controlled to below 800°C to prevent phase transition from α to β-phase, and excessive oxidation that can weaken PFM bonding.[47] Ceramics must also be matched to the low thermal expansion coefficients of the titanium (8-9.4X10_6/_C). Atsu and Berksun[48] showed it requires an argon atmosphere compared with a conventional vacuum to produce stronger metal-porcelain bonding. In another study by N. Nieva et al[49] it has been observed that the effect of sandblasting is decisive in the improvement of bond strength. Furthermore, the anodized layer probably produces an improvement of the chemical bond. In our study, for the uniformity of the samples only air abrasion was carried out.

The results of this study revealed that the bond strength of the specimens of all the three groups were higher than the recommended minimum value of 5.625 N, established by German Standard Institution DIN 13.927.[50]

Apart from bond strength, even the type of bond failure was noted. The higher the amount of ceramic body remaining on the metal surface, the higher the adhesion of ceramic to metal, the higher the fracture energy and the lower the risk of ceramic fracture would be in the clinical setting as stated by Wagner 1993[51] and Lavine 1966[52]. In our study, most of the specimens in all the groups showed cohesive failure. Cohesive failure within the porcelain is the most desirable bond failure mode[5], indicating a strong bond between the oxide layer and both the metal and ceramic. Since after debonding high amounts of ceramic remained on the alloy surface, it may be concluded that a strong bond was created at the interface.

Since this study had an in vitro design, the DMLS and TiLite alloy should also be evaluated in the oral cavity. Also, it is recommended to assess the marginal fit of copings fabricated by this technique. The bonding interface should be evaluated using scanning electron microscope and energy-dispersive X-ray spectroscopy in future studies.

Conclusion

Within the limitations of this in-vitro study, the following conclusions were drawn:

- According to the 3-point bending test results, inspite of statistically significant differences between metal–ceramic bond in all groups, the bending strength values of were within the acceptable levels of 5N as given in German Standard Institution DIN 13927.[50]
- The mean flexural bond strength of the Casted Co-Cr group was higher compared to DMLS-Co-Cr and Casted-TiLite groups.
- The metal-ceramic bond strength of the specimens, Casted-TiLite alloy, cover the lower acceptable limit of 5N, in accordance to DIN 13927; compared to the other two groups.
- Cohesive bond failure percentage was more in all the three groups, suggesting an acceptable bond between metal and ceramic.

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