A comparative evaluation of the effect of various surface treatments on the bond strength of ceramic fused to direct metal laser sintered cobalt chromium alloy- an in-vitro study

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ABSTRACT:

Objective: The purpose of the present study was to compare the influence of various surface treatments on the bonding surface of metal laser sintered Cobalt Chromium alloy cylinder and also to analyse the mode of failure in each category. The objectives were to evaluate and compare the bond strength of ceramic over Direct Metal Laser Sintered Cobalt Chromium alloy disc without surface treatment (Group I) and also with various surface treatments including sandblasting (Group II), acid etching (Group III) and laser ablation (Group IV).

Method: Forty samples of Cobalt Chromium cylinders of dimension 12 ×5 mm were fabricated using direct metal laser sintering. The samples were divided into four groups (N=10) samples each based on the surface treatments. After surface treatment, the samples were veneered with porcelain on the treated bonding surface. The shear bond strength test was done using Instron at crosshead speed 0.5 mm/min. The result were statistically analyzed using one way ANOVA Post hoc test followed by Dunnet t test ($p<0.05$). The mode of failure was also analyzed using Scanning Electron Microscopy.

Result: The mean bond strength of Group II and Group IV were greater than all other groups. The least bond strength was observed for Group III. The SEM analysis showed a cohesive failure for Group II and IV and mixed failure for Group I and Group II.

Conclusion: Laser ablation and sandblasting are effective surface treatments for bonding porcelain to Direct metal laser sintered Co-Cr coping.

Key words: Bond strength, Direct Metal Laser sintering, Laser, Porcelain fused to metal crown, Surface treatment.

1. INTRODUCTION

Metals are one among the most extensively used biomaterial in dentistry for many centuries. Modernization during the 20th century, have brought An exemplary transformation in dentistry by the inflow of new alloys that are economical than precious alloys and superior in properties that are more suitable for specific applications.[1] This casting techniques accredited the initiation of porcelain fused to metal restorations in 1950s which have been playing an important role in restorative dentistry since then because of greater colour stability and also the strength provided by the metallic framework.[2,3] The introduction of lost wax technique by W H Taggart in 1907 brought a major change in dentistry and since then this technique is being for casting base metal alloys. The procedure is technique-sensitive and in order to overcome the perplexity in casting, newer methods of processing these alloys has been researched prompting the introduction of direct metal laser sintering in dentistry.[4,5] Direct Metal Laser Sintering (DMLS) was introduced to meet the objective of producing metal parts directly from Computer Aided Designing data.[6] The initial Direct Metal Laser Sintering systems used either carbon dioxide laser or Yttrium Aluminum-Garnet lasers for laser machining. EOSINT M 270, the currently used system uses a 200 Watt Ytterbium fiber laser for the fabrication of crown and bridges. The high-power laser beam melts a bed of metal alloy powder by following a predetermined path layer by layer. This techniques directly fabricate, layer-by-layer, physical models from 3-D solid models produced in Computer Aided Designing. The dental restorations are prepared from biocompatible cobalt-chromium base metal alloys called as super alloys. The composition of this alloy does not contain tungsten and has lower molybdenum content, compared to the composition of the cobalt-chromium alloy for casting. The two variants of DMLS technique is powder bed and powder deposition method. In the powder bed method, the powder dispenser piston raises the powder supply and then a recoater arm distributes a layer of powder onto the powder bed. A laser then sinters the layer of metal powder. In the powder deposition method, the metal powder is contained in a hopper that melts the powder and deposits a thin layer onto the build platform. In both methods, after a layer is built, the build piston lowers the build platform and the next layer of powder is applied. Reproduction of complex geometry and post production finishing of restorations is efficient with laser sintering.[7] Considerable financial savings and reduced manufacturing time span is the greatest advantage of the Direct Metal Laser Sintering. Since the components are built layer by layer, it is possible to design internal features and passages that could not be cast or otherwise machined. The success of a metal ceramic restoration depends on the right choice of material and the bond between the porcelain and the metal substrate.[8] In an endeavour to improve the bond strength between metal and ceramic, various surface treatment modalities have been studied by many researchers on metal copings fabricated using conventional casting technique.. These include preoxidation of metal before ceramic application, application of bonding agent, air borne particle abrasion, degasification, surface modification with the help of carbide burs and diamond mounting tips, acid etching and laser ablation.[9-11] Various literatures have reported on internal fit and marginal fit of crowns and bridges fabricated using DMLS Co-Cr.[12,13,14] A research was conducted on the clinical acceptability and performance of DMLS Co-Cr bridges and concluded that porcelain veneer chipping is one of the reasons for the failure.[2] Hence the aim of the present study was to find the efficiency of direct metal laser sintered coping to bond with porcelain by using various surface treatments like sand blasting with Aluminium oxide, Etching with Nitric acid and Hydrochloric acid and Laser ablation.

2. MATERIALS AND METHODS

Forty samples of Cobalt Chromium cylinders of dimension 12 x 5 mm were fabricated using Direct Metal Laser Sintering. The bonding surfaces of DMLS Cobalt Chromium cylinders were exposed to various surface treatments to assess the bonding efficiency of ceramic over the same. The samples were divided into four groups of ten samples each based on the surface treatments and the groups were **Group I** – without any surface modification, **Group II** – surface treatment with 110 micron size aluminum oxide, **Group III** – surface treatment done by acid etching using hydrochloric and nitric acid , **Group IV** – surface modification done by laser ablation. For the fabrication of Cobalt Chromium cylinders, Direct Metal Layer Sintering technique was done using EOSINT M 270 system. EOS Cobalt Chrome SP, a multi-purpose Cobalt Chrome-Molybdenum based super alloy powder was used for the fabrication of 12 × 5 mm Cobalt Chromium cylinders. For this, a CAD data was created using an optical scanner and CAD software. This data was then transferred to the Direct Metal Laser Sintering unit which was equipped with a 200W Ytterbium- fiber laser that could finely focus a short wavelength laser beam to a diameter of 100 µm. The use of a short wavelength ensured high absorption of energy into the metal powder, so that build speed was optimized. After manufacturing Cobalt Chromium cylinders, they were ground using 1200-grit sandpaper. All substrates were ultrasonically cleaned for 10 minutes and rinsed in distilled water to remove the contaminants. For each group, specific surface treatments were done on the bonding surface.

Surface treatment of bonding surface of the samples

Group II samples were treated using air borne particle abrasion with 110 micron Aluminium oxide particles at 2 to 3 bars air pressure for 10 seconds from a distance of 30mm using a special holder. Group III samples were acid etched by immersing the specimens in 35% by weight aqueous solution of Hydrochloric acid and concentrated Nitric acid in glass container and kept for 30 minutes. The samples were subjected to ultrasonic cleaning using distilled water for 10 minutes and then rinsed with distilled water. The group IV Cobalt Chromium specimens were positioned on a mounting jig in the laser machine and the bonding surface was irradiated by the linear movement of a glass fiber of near diffraction limited Q- switched Nd : YAG laser at a power setting of 0.5 W and frequency levels of 120 mJ with 10-Hz frequency for 2 min. All the surface treated samples were analyzed under Scanning Electron

Microscope under 4000 x and 2000x magnification to assess the quality of surface modification. The specimens were ultrasonically cleaned for 1 minute in distilled water and air-dried for 30 seconds after the specific surface treatments before layering the porcelain. The specimens were analysed using Scanning Electron microscope for the surface changes as shown in figure (1a, 1b,1c & 1d)

Veneering the Cobalt Chromium samples with porcelain

Porcelain layering was done on the bonding surface of all metal specimens. 5mm diameter and 3mm height porcelain discs were layered over the metal surface. Two layers of opaque, dentin porcelain thickness of 0.5 mm, enamel porcelain thickness of 2.4 mm and firing temperature of 880^0 C was used for the fabrication of porcelain discs. A second application of this porcelain layer and another porcelain firing cycle was needed to compensate for the contraction generated during the first firing cycle, allowing achievement of the final dimensions. A metal plate of 3 mm thickness with a 5mm diameter hole was fabricated to ensure the dimensions of the disc. A vernier caliper was also used to confirm the measurement of the specimens. Direct Metal Laser Sintered Cobalt Chromium cylinder was inserted in to the acrylic with the veneered surface facing outward for the ease of bond strength test. Shear bond strength test was done with a universal testing machine at a crosshead speed of 0.5 mm/min. Specimens were loaded to failure by applying a shear force at the veneer-alloy interface. The shear bond strength was analyzed with the formula $\delta = P/\pi r^2$ where δ is SBS [MPa], P is load at failure [N], π is 3.14, r is radius of veneering disc [1mm]. The data were statistically analyzed using ANOVA Post hoc test followed by Dunnet t test. Scanning Electron Microscopic analysis was performed to assess the mode of failure. The result of the bond strength was coupled with the failure analysis. For this, two representative specimens from each group were selected for examination of the metal-ceramic interface under SEM. These specimens were ultrasonically cleaned in distilled water for 10 minutes prior to the making of SEM analysis. The specimens were Gold spluttered and observed under 2000 x magnification to determine the mode of failure. The debonded metal surface of the samples was viewed under SEM at 2000 x magnification to determine the mode of failure. All failures were classified into adhesive failure (no remnants of porcelain veneer on DMLS metal core), cohesive failure in porcelain veneer (only the remnants of porcelain veneer seen on the fractured surface), mixed failure (both adhesive and cohesive failures detected on the fractured surface).

3. RESULT AND STATISTICS

The data is expressed in Mean and Standard deviation(Table 1, graph 1). Analysis was done by SPSS (16.0) version software. One way ANOVA (Post hoc test) followed by Dunnet t test applied to find statistical significant between the groups. P value less than 0.05 ($P<0.05$) considered statically significant at 95% confidence interval. The mean value of shear bond strength of group I specimens to porcelain were 25.11 MPa which was adequate as per ADA specification. Group I was statistically significant with Group II, III and IV. The mean value of shear bond strength of group II samples to porcelain was higher with a value of 32.09 MPa which was statistically significant with Group I and III. Shear bond strength value of Group II it did not show any statistical significance with Group IV. The mean value of shear bond strength of group III samples to porcelain were lower with a value of 9.36 MPa and were statistically significant with the other groups. The mean value of shear bond strength of group IV samples to porcelain were higher with a value of 32.36 MPa was statistically significant with the group I and group III samples and statistically insignificant with group II samples. Based on the study, it was found that the most effective surface treatment is sand blasting followed by laser ablation. The bond strength was found to be the least in group III (acid etched) and it cannot be advocated for surface modification and bonding with porcelain. The mode of failure was also assessed to evaluate the most effective surface treatment. The scanning electron microscopic image of group I and III (Figure. 2a & 2c) exhibited the presence of ceramic particles on few areas of metal surface indicating mixed type of failure. The SEM image of group II and IV (Figure.2b & 2d) samples showed the presence of ceramic throughout the debonded surface indicating cohesive type of failure.

Table-1: Comparison of Mean shear bond strength (MPa) of different groups

Groups	Shear Bond Strength (MPa) (MEAN±SD)	Group Comparison	P value
Group-I	25.11±1.74	G-I with G-II, III, IV	
Group-II	32.09±2.30[a]	G-II with G-I, III	
Group-III	9.36±8.13[a,b]	G-III with I, II,IV	**0.001**
Group-IV	32.36±2.11[a,c]	G-IV with I, III	

(a,P<0.001 significant compared to group-I with II,III and IV; b, P<0.001 significant compared to group-II with others I and III; c ,P<0.001 significant compared to group-III with I, II, IV P<0.05 not significant compared group-II with IV)

Graph-1: Comparison of mean shear bond strength (MPa) of different groups

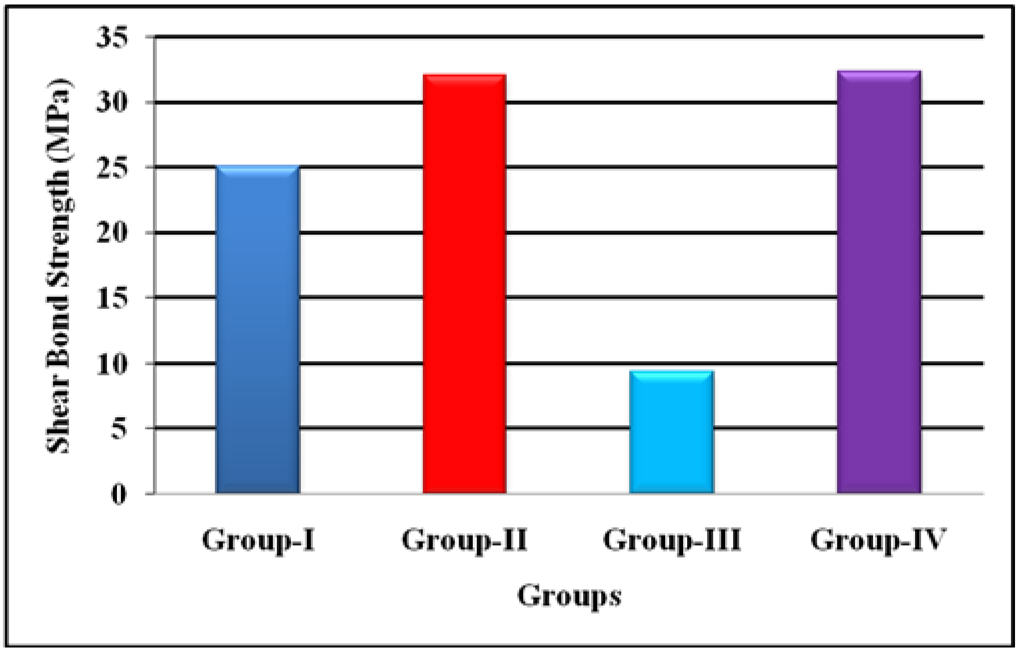


Fig. 1a - Bonding surface of Group I DMLS disc without surface treatment.

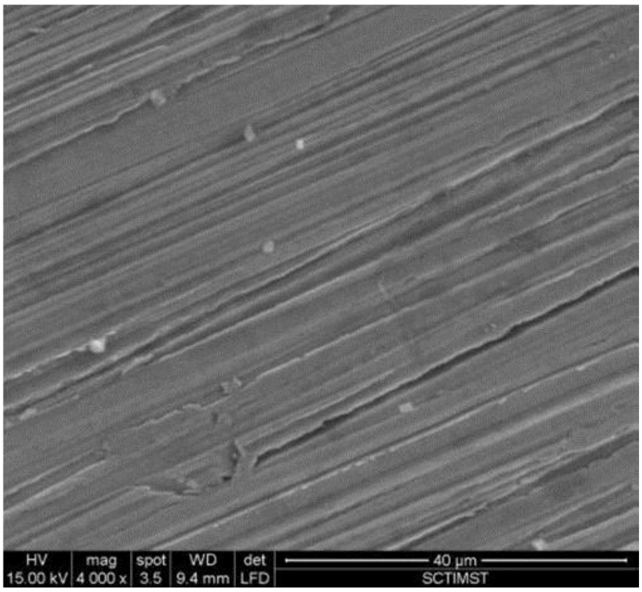


Fig. 1b - Bonding surface of Group II DMLS disc after sandblasting.

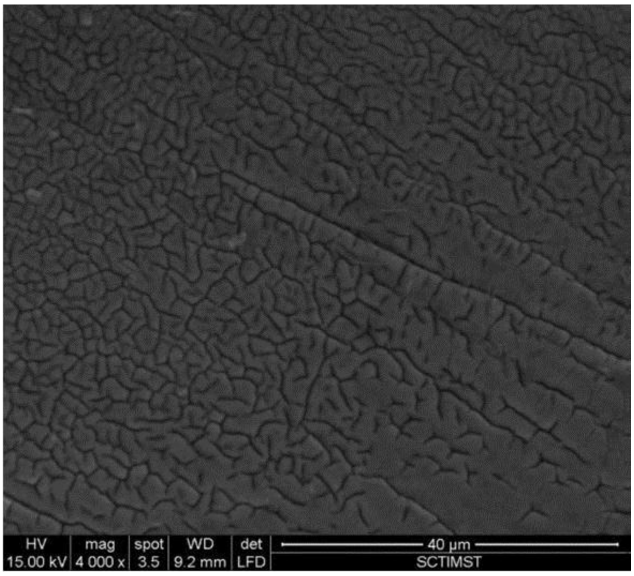


Fig. 1c - Bonding surface of Group III DMLS disc after acid etching.

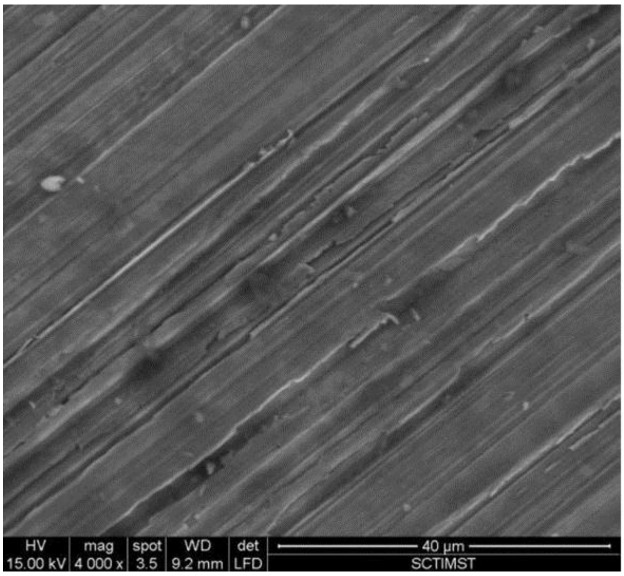


Fig. 1d - Bonding surface of Group IV DMLS disc after laser ablation.

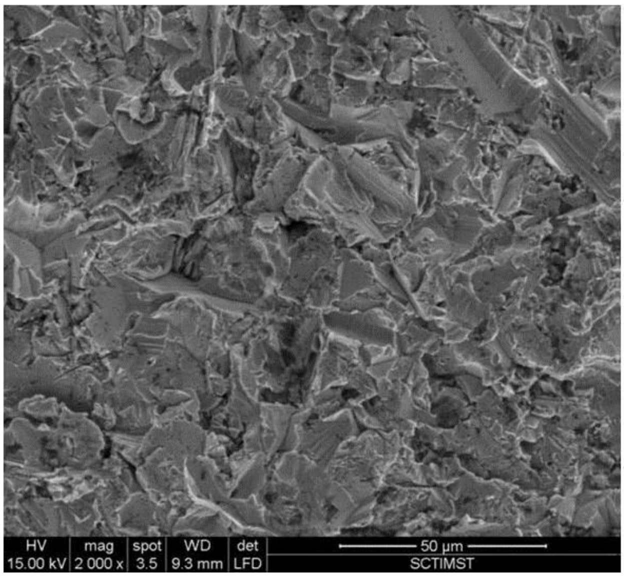


Fig.2a- Surface of Group I DMLS disc after debonding

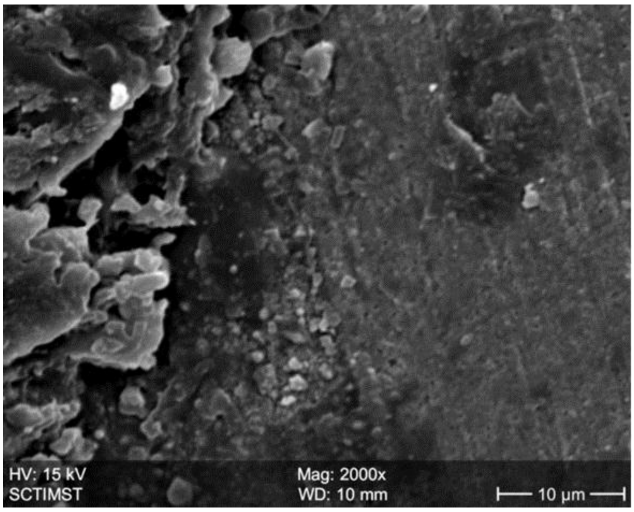


Fig.2b- Surface of Group II DMLS disc after debonding

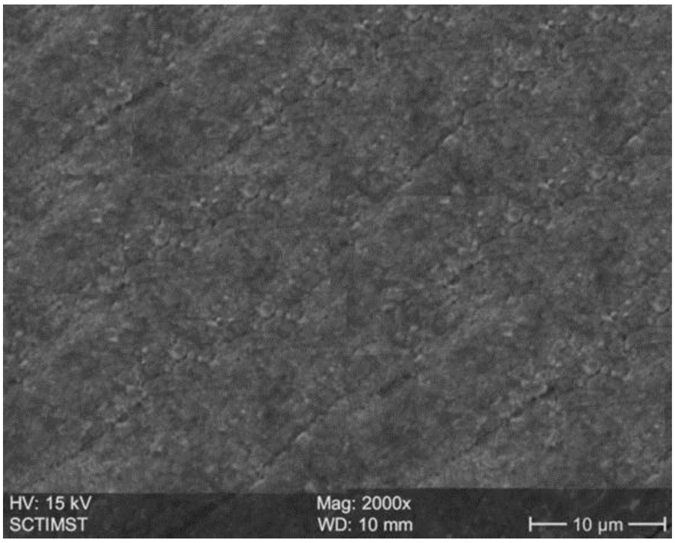


Fig.2c- Surface of Group III DMLS disc after debonding

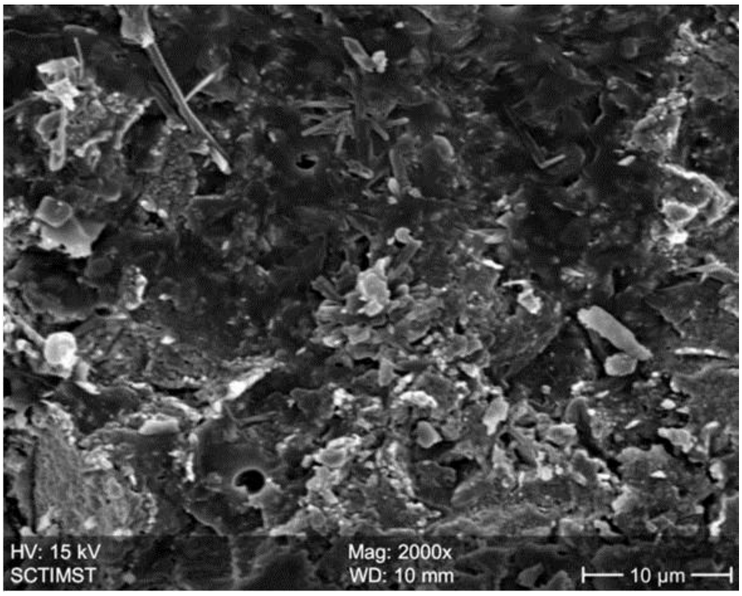
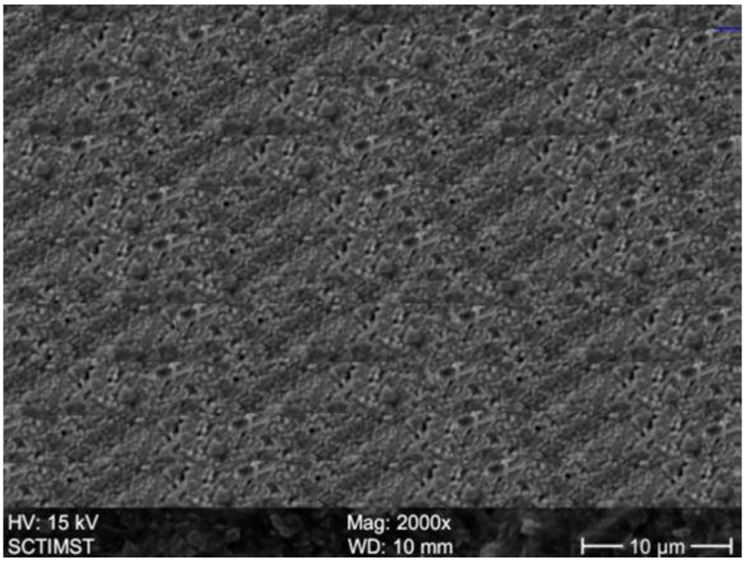


Fig.2d- Surface of Group IV DMLS disc after debonding



4. DISCUSSION

Laser-based layer manufacturing technique is a recent development in dentistry which has the advantage of producing metal products of high density and excellent mechanical properties.[6] The versatility, improved material properties, and shortened product development cycles has assured the success of metal laser sintered crowns in dentistry.[15-17] Based on this study, GROUP I samples were not surface treated and was considered as the control group. The current standards, ANSI/ADA Specification No. 38 (2000) and IRAM-ISO Standard 9693 (1999), for evaluation of the metal–ceramic bond, the minimum acceptable bond strength is

25MPa.[7,18,19] The samples in this group showed a mean bond strength value of 25.11 MPa. In Direct metal laser sintered Cobalt Chromium, there is an inevitable degree of surface roughness as it is built up from powder. The roughness is typically between 4.5-6.3 Ra for every 20 µm, which improves the bond strength. Wanger et al stated that there is a direct co relation between roughness and bond strength[20]. The Coefficient of Thermal Expansion difference also helps in the bond strength of Direct Metal Laser Sintered Cobalt Chromium alloys to porcelain. The Coefficient of Thermal Expansion of the metal substructure should be approximately 0.5×10^{-6} higher than that of the applied ceramic.[21] Coefficient of Thermal Expansion of Ceramco II ceramic system is 13.5×10^{-6} and the Direct Metal Laser sintered Cobalt Chromium alloy is $14\text{-}14.4\times10^{-6}$.[22] On inspection, the CTE of ceramic and Direct Metal Laser sintered Cobalt Chromium is found to be approximately 0.5×10^{-6} which could have been one of the reasons for enhancing the bond strength of the samples in this group. In Direct Metal Laser sintering the quick solidification after melting leads to fine and homogenous micro structure of the alloy thereby lowering the porosity. This can also attribute to the strong bond between Direct Metal Laser Sintered Cobalt Chromium and porcelain. Therefore it could be ascertained that the above mentioned factors has lead to the satisfactory bond strength of Direct Metal Laser Sintered Cobalt Chromium specimens even without surface modifications. Group II samples in which the Direct Metal Laser Sintered Cobalt Chromium cylinders were treated with Aluminium oxide of 110 micron in size at 2 to 3 bars air pressure for 10 seconds showed a mean bond strength of 32.09 MPa which was statistically higher than all other groups . It has been estimated that sandblasting produces a micro retentive roughness and thus increases the total surface area of a metallic substrate by up to 6.5 times which in turn increases the metal-ceramic bond strength of dental alloys[23]. The air borne particle abrasion increases the metal surface energy and result in improved wettability of opaque ceramic and consequently the bond strength through micro mechanical bonding.[24] This could be the reason for the highest bond strength for group II samples. Group III in which the Direct Metal Laser Sintered Cobalt Chromium cylinders were treated with 35% Hydro chloric acid and concentrated Nitric acid showed mean bond strength of 9.36 MPa which was statistically lower than all other groups. The Direct Metal Laser Sintered Cobalt Chromium which is processed with a layer thickness of 20 µm has dendrites in their microstructure that extends vertically to the adjacent layer like a Honey comb structure with 1µm length. Thus microstructure is more homogenous compared to the cast microstructures. So even if the acid etching removes a layer of DMLS Cobalt Chromium specimen, a homogenous layer still remains in the metal surface. The grain size of the alloy is 0.3-0.6 µm. Hence the roughness created on the laser sintered metal surface due to the disintegration of the grains might be smaller in size. When acid is applied on Cobalt Chromium, alloy acid releases Cobalt from the alloy and the Chromium content is increased at the surface. This Chromium will increase the thickness of oxide layer on the metal surface since it forms oxide layer in the initial step of oxidation itself.[21] This heavy oxide layer and the minimal roughness created due to the small grain size could be the reason for decrease in bond strength of acid etched Direct Metal Laser Sintered Cobalt Chromium. Group IV in which the Direct Metal Laser Sintered Cobalt Chromium cylinders were laser ablated showed a mean bond strength of 32.36 MPa which was statistically higher than group I(control),group III(acid etched). For laser ablation Nd :YAG laser is used. Nd :YAG laser has comparatively lower wave length in comparison with other laser systems. Santos et al stated that the absorptivity of metal increases with decrease in wave length and a higher melting depth can be observed in the same powder density by using a Nd:YAG laser since they use optical fibers to guide them.[6] This study is in agreement with the study conducted by Kim et al were they stated that Laser etching of metal surfaces using an Nd /YAG laser was effective in improving bond strength.[11] When laser interacts with metal, the laser energy is absorbed primarily by free electrons. The absorbed radiation energy in the skin layer involves thermalization within the electron sub-system. Due to the electron thermal diffusion, energy transfers to the lattice and heat is transported into metal. Generally, depending on the fluence of the laser pulses, a series of effects such as melting, vaporization of the molten materials, dissociation and ionization of the vaporized material, etc., can be generated on the target. The morphological changes, such as micro cracking and nano grains, were mainly the consequence of rapid heating and cooling of the metal target surface layer also, minor fraction of material is ejected at the periphery in this case. A direct consequence of these effects is the increase in the surface roughness of the target. Laser and electron-beam thermal treatments could be used for modification of the microstructure of titanium surfaces without contamination for providing optimal roughness.[11,20] The shear bond strength of laser-ablated surface is statistically lower than that of sand blasted group. This may be due to the presence of deep fissures that are created during laser ablation. This is in agreement with the study of Wagner et al which stated that surface roughness can weaken the ceramic metal inter face by causing stress concentration and the roughness can also lead to incomplete contact between metal and ceramic.[20] Considering the mode of failure, the bond between metal and porcelain should be greater than the cohesive strength of the porcelain. Results of this study showed cohesive failure for sandblasted and laser treated surfaces. Mixed failure mode needs opaque composite for repair and may be difficult to be optimally repaired within the operatory. Hence based on the bond strength and mode of failure, sandblasting and laser surface treatment can be considered effective for porcelain fused to metal crown using a DMLS coping. Even though sand blasting provides the maximum bond strength , studies have stated that air borne particle ablation can contaminate the metal surface with alumina particle.[25] The presence of such embedded fragments adversely affects the bond strength of metal-ceramic and metal-resin systems, decreasing the mechanical interlocking and inhibiting the chemical bonding of porcelain with metallic oxides.[26] Hence in clinical cases with high masticatory force, the likelihood of porcelain fracture is higher. Gaggl et al [27] and Cho and Jung [28] reported that laser treatment would be an effective method for producing surface roughness without any contamination. Hence a controlled laser treatment can be considered as an effective alternative to airborne-particle abrading for enhancing the bond strength of porcelain to metal especially in areas of high masticatory force.

5. CONCLUSION

Thus from the present study, it could be concluded that Direct Metal Laser Sintering technique is one of the best of its kind for obtaining core for metal ceramic restorations. The strongly recommended surface treatment based on the present study includes the use of sand blasting and laser ablation which could be used for obtaining a clinically successful restoration. On analyzing it could be found that the present study is not without limitation and requires further extended research. The study performed is an in- vitro study and does not simulate the oral environment which is very complex. To assess the complete clinical success of a restoration, an in-vivo study has to be done. The thickness and composition of oxide layer for Direct Metal Laser Sintered Cobalt Chromium alloy which can highly influence the bond strength. Hence further researches should be performed using Energy Dispersive Spectrometric Analysis (EDS) for surface analysis. The present study can be extended and clinically applied after assessing the application of various other lasers on the bonding surfaces of Direct Metal Laser Sintered Cobalt Chromium copings.

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