

IN-VITRO COMPARATIVE STUDY TO EVALUATE BOND STRENGTH OF PORCELAIN ON METAL SUB-STRUCTURE FABRICATED BY SELECTIVE LASER MELTING (SLM) TECHNIQUE AFTER APPLICATION OF DIFFERENT SURFACE TREATMENTS

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ABSTRACT

Introduction: Metal-ceramic bond strength is one of the essentials that must be studied due to recent developments in dentistry.

Objectives: This study aimed to evaluate the ceramic bond strength with metallic sub-structure made of chromium-cobalt alloy and fabricated by selective laser melting (SLM) technique after applying different surface treatments.

Material and methods: 40 metallic strips of Co-Cr alloy manufactured by SLM technique were divided into two main groups, A and B, which were again divided into 4 sub-groups as follows: A1 – natural surface roughness maintained ($n = 10$); A2 – natural roughness with a bonding agent application ($n = 10$); B1 – sandblasted surface ($n = 10$); B2 – sandblasted surface with a bonding agent application ($n = 10$). A surface roughness measuring test was carried out to obtain Ra-Rz values. Three-point bending test according to ISO 9693 standards was performed to determine the metal-ceramic bond strength in MPa. Fracture failure mode was determined by using an optical microscope.

Results: Student's *t*-test was used to compare among the mean bond strengths of sub-groups, with a significant difference observed only between A1 and A2 and between A2 and B2 sub-groups. Mann-Whitney test was applied to compare among the mean ranks of the failure modes, with a significant difference noticed only between A1 and A2 sub-groups.

Conclusions: The bond strength in all the studied sub-groups exceeded the required minimum 25 MPa, according to ISO 9693 standards. The bonding agent had a minor effect when applied on sandblasted surfaces, while it had an important effect when applied on a natural surface roughness.

Key words: Co-Cr alloy, metal surface roughness, metal-ceramic bond strength, selective laser melting (SLM) Co-Cr alloy.

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INTRODUCTION

The success of metal-ceramic prosthodontics is related to the metal-ceramic bond strength, where the separation of porcelain out of the metal is one of the most common mechanical problems that could be encountered within dental practice [1].

There are different techniques for fabrication of metal structures, such as lost wax technique, which is one of the most widely used techniques in dentistry [2]. However, nickel-chromium alloy that is extensively used in the lost wax technique showed unfavorable biological effects and hypersensitivity comparing with the other alloys due to the presence of nickel [3].

Therefore, the use of chromium-cobalt alloy has become an alternative to nickel-chromium alloy in the field of metal-ceramic restorations, as it is safer and does not cause toxic effects and allergic reactions in patients who are allergic to nickel [2, 3].

Recently, a new technique for fabricating metal structures has appeared, called 'selective laser Melting' (SLM) technique [4]. The SLM technique belongs to additive manufacturing process, in which the metal is formed from a 3D CAD file by selective melting of metal particles [4]. It is considered one of the innovative modern techniques after selective laser sintering (SLS) technique [5].

SLM technique emerged as an alternative to the traditional casting technique, as many problems and errors associated with traditional casting process were avoided [6]. In addition to improving the mechanical properties of the formed metal structure [5], the surface of metal manufactured by SLM technique has a natural roughness. This is permanently formed roughness on the surface of metal manufactured with this technique, as it may require minimal surface treatment [4].

A previous study had shed a light on the effect of the natural roughness resulting from SLM technique in improving bonding strength [4], where the positive role of natural roughness of SLM-made metal in increasing the strength of metal-ceramic bond was emphasized.

The metal-ceramic bond is basically achieved through the following basic factors, such as chemical bonding, mechanical interlocking, Van der Waals forces, and the compression resulting from the difference in coefficient of thermal expansion between the metal and porcelain [7].

Three-point bending test has been adopted by several previous studies investigating the issue of metal-ceramic bond strength [4, 8-10]. The three-point bonding test used in the present study was according to ISO 9693 standards [11].

One of methods used to improve the bond strength is to expose the metal surface to aluminum oxide particles by sandblasting in order to roughen the metal surface and to increase the bond strength [12]. That allows to increase the surface wettability, in particular the contact area between the metal and the ceramic, which can significantly improve the strength of mechanical bonds [13].

A bonding agent has also been introduced for use in order to improve the metal-ceramic bond strength through compensating the difference in the expansion coefficients between the metal and the applied ceramic, which blocks escaping metal oxides [14, 15]. On the other hand, authors found that the bonding agent had a minor influence in increasing the strength of the connection [16-18].

OBJECTIVES

The aim of the present study was to evaluate the ceramic bond strength with metallic sub-structure made of chromium-cobalt alloy, and fabricated by SLM technique after applying different surface treatments.

MATERIAL AND METHODS

The total sample consisted of 40 metal strips made of Co-Cr alloy using SLM technique. The strips were designed according to ISO 9693 standards, with 25 × 3 × 0.5 mm dimensions. SOLIDWORKS 3D CAD software standard was employed for this purpose. After designing the strips, the Co-Cr alloy was used in powder form (Scheftner, Starbond CoS Powder 30, Germany). A selective laser melting machine (SISMA MYSINT 100 Machine, Italy) was used. Parameters needed for the melting process were adjusted according to the manufacturer's instructions. Inclination angle (0) between the platform

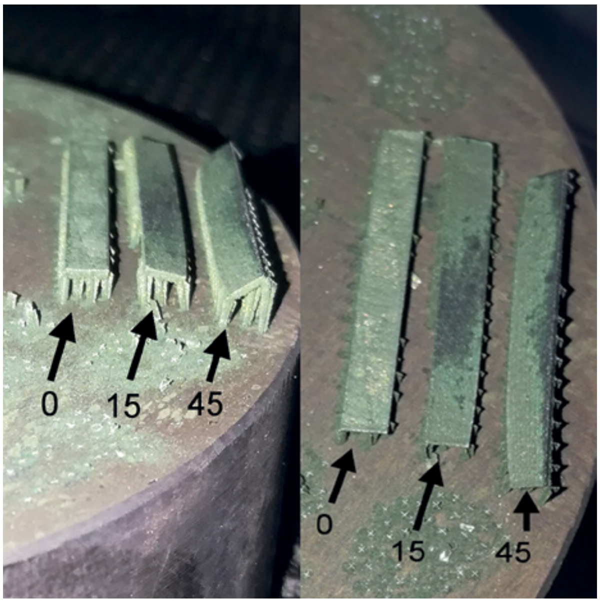

FIGURE 1. Three experimental specimens manufactured by selective laser melting (SLM) technique with different inclinations (0-15-45) between the strip and the metal platform

and the manufactured strip was used to prevent deformation, since two extra experimental strips were carried out with different inclination angles (15-45), where deformation in the strips was observed after fabrication process (Figure 1). After the strips were manufactured, they were all entered into a special oven (Nabertherm, Germany) within an inert atmosphere due to argon gas present as protective gas to prevent ignition, according to the manufacturer's instructions. The thickness of each metallic strip (0.5 mm) was confirmed with a dial caliper at several points (6 measurements for each specimen).

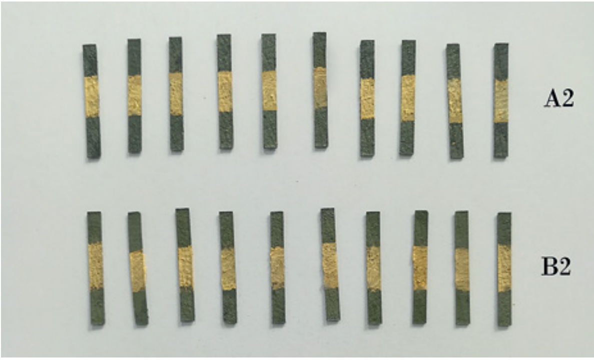

FIGURE 2. Application of bonding agent in subgroups A2 and B2

FIGURE 3. Whole studied sample (A1, A2, B1, B2) after applying the porcelain

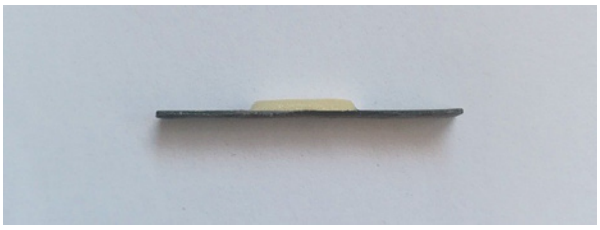

FIGURE 4. One of the metal ceramic strips after application of porcelain

A Vernier caliper was used to confirm width (3 mm) and length (25 mm).

The sample (40 metal strips) was randomly divided into two equal main groups (20 strips per group), and each main group was randomly and equally divided into two sub-groups (10 strips per group) as follows:

Main group A:

1. Sub-group A1: the natural surface roughness was maintained without applying a bonding agent (10 strips).

2. Sub-group A2: a bonding agent was added on the natural surface roughness (10 strips).

Main group B:

1. Sub-group B1: the surface was treated by means of airborne-particle abrasion with aluminum oxide particles (10 strips).

2. Sub-group B2: a bonding agent was applied on the sandblasted surface (10 strips).

Metal surface of the specimens from the main group (B) was treated by means of airborne-particle abrasion with aluminum oxide (Al_2O_3) particles, with a particle size of 110 µm at 2 bar at an approximate distance of 10 cm between the nozzle and the surface for 10 seconds.

For surface roughness measurement, 20 strips were randomly selected to measure the surface roughness as follows:

- 10 strips from the main group A;
- 10 strips from the main group B.

The measuring device used was a perthometer S2 (Mahr, Germany). In order to measure the roughness average (Ra) and mean roughness depth (Rz), six orthogonal measurements of 5.6 mm were taken in the central area, where the porcelain was planned to be applied afterwards. A custom-made mold was adjusted to fit the strip's dimensions to prevent instability during the measuring process, and hence to obtain better readings.

For porcelain layering, all strips were cleaned by Steam jet for 15 seconds. Bonding agent (Bredent GmbH, Germany) was applied in one layer on 20 strips divided randomly in the two sub-groups A2 and B2 (Figure 2). Opaque & Dentin (GC Initial® Metal Ceramic) were used for the application on each specimen (Figure 3). The place where the porcelain was applied in the middle of the strip (8 mm in length and 3 mm in width) was determined by using a sharp blade. Opaque ceramic (Wash Opaque & Paste Opaque) was applied in two layers, according to the manufacturer's instructions. Then the dentinal porcelain was applied in two stages to compensate for any possible shrinkage of the porcelain. The surface was subsequently polished (CeraMaster® Coarse Polishers, Shofu Inc.), and the specimen was ready to be tested by three-point bending test (Figure 4).

The entire required metal-ceramic thickness (1.5 mm) was ascertained by means of a dial caliper at six random points from the surface.

Bond strength test according to ISO 9693:

A universal testing machine was used (IBMU4 – 1000, S.A.E. IBERTEST; Spain). In order to perform the three-point bending test on the strips to find out the bond strength values estimated in MPa, force (in Newtons) was loaded in the center of the strip at a speed of 1.5 mm/min until separation occurred, which indicated failure (Figure 5). Bond strength value (MPa) was obtained by multiplying the force (F_{fail}) before de-bonding (failure load) with the value of constant (κ) as a function of metal substrate thickness and Young's modulus of the metallic material. (κ) value determinant was obtained from ISO 9693 standards. The calculation formula was as follows:

$$\Sigma = \kappa \times F_{fail}$$

The failure mode was observed and determined after detaching the ceramic layer manually by using an optical microscope (Ailabuk, A & E Lab, Co. Ltd., UK) with 4× magnification (Figure 6). The failure mode was classified according to Akova *et al.*, as adhesive failure, cohesive failure, and mixed failure [10].

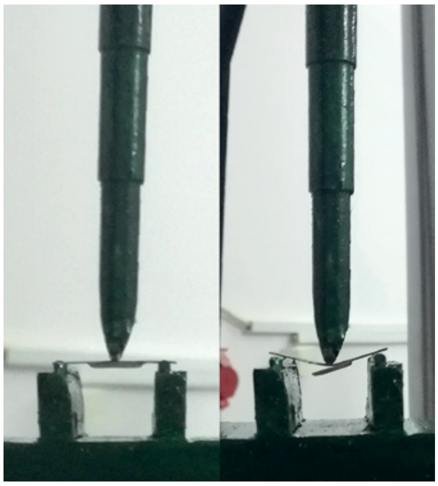

FIGURE 5. Three-point bending test device. Before applying the force (on the left). After applying the force (on the right)

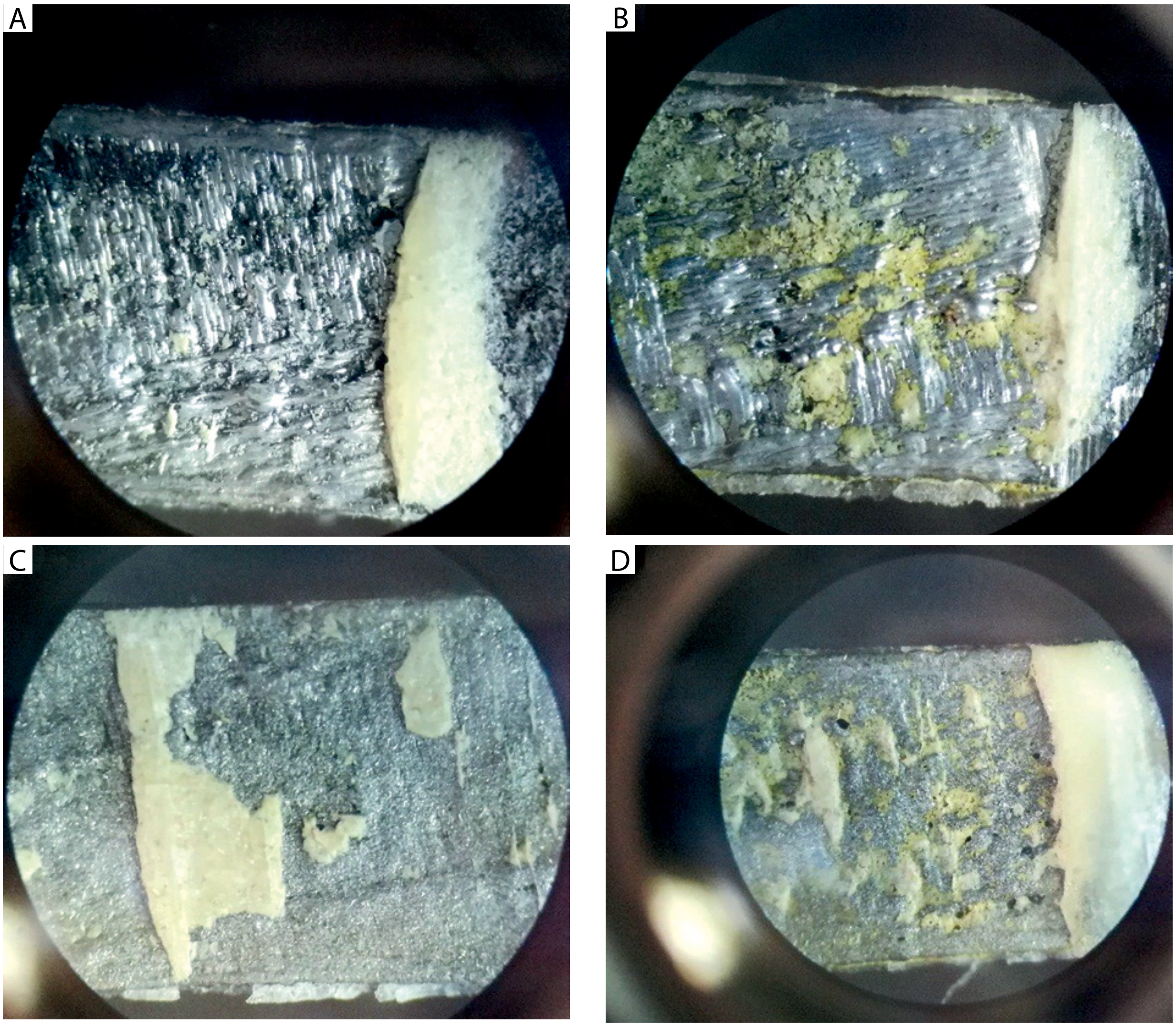

FIGURE 6. Fracture surface of specimens from the four subgroups under the microscope 4× magnification; **A**) indicating to the fracture surface of specimen from subgroup A1; **B**) indicating to the fracture surface of specimen from subgroup A2; **C**) indicating to the fracture surface of specimen from subgroup B1; **D**) indicating to the fracture surface of specimen from subgroup B2

RESULTS

SURFACE ROUGHNESS MEASUREMENT

The perthometer measuring device used in this study is based on ISO standards (DIN EN ISO 4287, ASME B46.1), and considers irregular roughness unreadable. Therefore, the specimens that maintained natural roughness were illegible due to irregularity of the natural surface roughness formed with SLM technique. The roughness measuring values of the sandblasted specimens were readable, as shown in Table 1.

THREE-POINT BENDING TEST ANALYSIS

The three-point bending test was performed for all the specimens, and the bond strength values (MPa) were obtained, as presented in Table 2.

Normality of data distribution was tested using Kolmogorov-Smirnov (KS), which showed that the distribution was normal for all tests, with *p*-value > 0.05 (therefore, applied parametric tests were applied), except for failure modes, in which the distribution was not normal with *p*-value < 0.05 (therefore, non-parametric tests were used).

Descriptive statistics of ceramic-metal bond strength are demonstrated in Table 3 and Figure 7. Student's *t*-test was applied to compare mean values of the ceramic-metal bond strength for the studied sub-groups, as shown in Table 4.

From Tables 3 and 4 can be seen that *p*-value was < 0.05 when comparing A1 with A2 sub-groups. Therefore, there was a statistically significant difference between the mean bond strength of the two sub-groups. Also, the mean bond strength of A2 sub-group was significantly higher by 60.06%. *P*-value was > 0.05 when comparing B1 with B2 sub-groups. Therefore, there was no statistically significant difference between the mean bond strength of the two sub-groups. The mean bond strength of B2 sub-group was higher by 16.80%. Also, *p*-value was > 0.05 between A1 and B1 sub-groups, and there was no statistically significant difference between the mean bond strength of the two sub-groups.

TABLE 1. Surface roughness measurements values of the sandblasted specimens

Ra (µm)	7.536	6.627	7.951	6.800	6.159	7.074	8.088	7.326	8.397	7.115
Rz (µm)	30.90	26.10	36.80	32.40	32.10	33.60	37.70	31.58	32.21	30.91

TABLE 2. Three-point bending test results of metal–ceramic bond strength (MPa)

Sub-group A1	A1 (1)	A1 (2)	A1 (3)	A1 (4)	A1 (5)	A1 (6)	A1 (7)	A1 (8)	A1 (9)	A1 (10)
	27.3	27.3	27.3	42.9	46.8	35.1	35.1	27.3	46.8	109.2
Sub-group A2	A2 (1)	A2 (2)	A2 (3)	A2 (4)	A2 (5)	A2 (6)	A2 (7)	A2 (8)	A2 (9)	A2 (10)
	85.8	70.2	62.4	74.1	54.6	74.1	70.2	74.1	70.2	70.2
Sub-group B1	B1 (1)	B1 (2)	B1 (3)	B1 (4)	B1 (5)	B1 (6)	B1 (7)	B1 (8)	B1 (9)	B1 (10)
	46.8	30.7	54.6	35.1	42.9	46.8	42.9	35.1	35.1	74.1
Sub-group B2	B2 (1)	B2 (2)	B2 (3)	B2 (4)	B2 (5)	B2 (6)	B2 (7)	B2 (8)	B2 (9)	B2 (10)
	62.4	74.1	46.8	31.2	42.9	54.6	50.7	50.7	50.7	54.6

TABLE 3. Descriptive statistics of ceramic-metal bond strength (MPa) in all sub-groups

Sub-group	Max	Min	Standard deviation	Mean
A1	109.20	27.30	24.76	42.51
A2	85.80	54.60	8.11	70.59
B1	74.10	30.70	12.69	44.41
B2	74.10	31.20	11.34	51.87

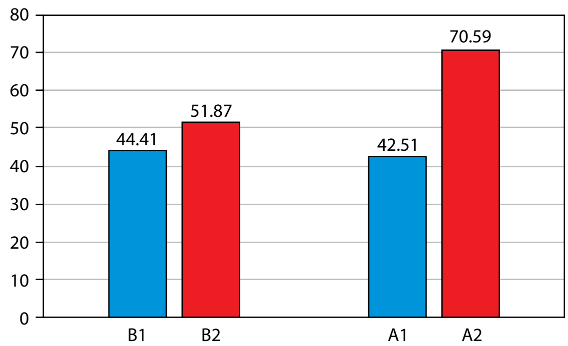

FIGURE 7. Descriptive statistic – mean bond strength of the studied subgroups

TABLE 4. Student's *t*-test results (MPa) of metal-ceramic bond strength mean values among sub-groups

Sub-groups	Difference in means	*t*-test	*p*-value	Result
A1 with A2	28.08	3.408	0.003**	Significantly different
B1 with B2	7.46	1.386	0.183 N.S.	Non-significantly different
A1 with B1	1.90	0.216	0.831 N.S.	Non-significantly different
A2 with B2	–18.72	–4.246	0**	Significantly different

The mean bond strength of B1 sub-group was higher by 4.47%. While p-value was < 0.05 after applying the bonding agent between A2 and B2 sub-groups, there were statistically significant differences between the mean bond strength of the two sub-groups. The mean bond strength of A2 sub-group was higher by 36.09%.

FRACTURE FAILURE MODE ANALYSIS

The fracture failure mode ranks of the studied sub-groups were obtained by using an optical microscope. as presented in Table 5. Mann-Whitney test was used to compare among the mean ranks of the failure modes. The test results are shown in Table 6. P-value was < 0.05, therefore, there was a statistically significant difference when comparing the mean ranks of the failure modes between the A1 and A2 sub-groups. There was no significant difference when comparing the mean ranks between the B1 and B2 sub-groups, with p-value > 0.05.

TABLE 5. Fracture failure modes of sub-groups

Sub-group	Mixed failure	Cohesive failure	Adhesive failure
A1	3	0	7
A2	10	0	0
B1	7	0	3
B2	8	0	2

TABLE 6. Mann-Whitney test results comparing mean ranks of fracture failure modes

Sub-group	Mean ranks	Mann-Whitney test	*p*-value	Result
A1	7	15	0.007 N.S.	Statistically significant
A2	14			
B1	10	45	0.739 N.S.	Statistically non-significant
B2	11			

DISCUSSION

In this study, many comparisons were made to better conclude average of metal-ceramic bond strength. As the metal sub-structure made of Co-Cr alloy was fabricated by the SLM technique, the porcelain was applied to the metal surfaces subjected to different surface treatments.

The alloy of Co-Cr used in this study was in powder form, as the metal parts manufactured by SLM are basically present as powdered metal for a selective laser melting process [5].

Co-Cr is a base alloy and more susceptible to oxidation than precious alloys during traditional casting process [8]. Co-Cr contributes to better chemical bonding with different types of ceramics [14]. In a previous study, it was found that the oxide layer was less in thickness in SLM technique compared with traditional casting technique [19, 20], and the thinner oxide layer faded when warming up in a furnace, while the thick layer caused weak bonding [21]. Henriques *et al.* indicated that the pre-oxidation process led the metal-ceramic bond strength to weaken [22]. In this study, the Co-Cr alloy was not subjected to pre-oxidation according to the manufacturer's instruction. Metal strips that make up the whole sample were designed using the following dimensions: 25 × 3 × 0.5 mm, according to ISO 9693 standards. Three-point bending test was conducted in this study, which has been approved by ISO 9693 since 1996. It is an accurate and high-sensitivity test, and has been applied in many similar previous studies [4, 9, 10, 12].

After the fabrication process, the metal strips were placed in a special furnace for heat treatment, which is necessary to release residual stresses. This furnace is equipped with argon (inert gas) as protective gas to prevent a flammable reaction, according to the manufacturer.

The metal strips were cleaned before applying the porcelain by steam jet, according to other similar studies [9, 10]. GC Initial® Metal Ceramic and Bredent Ceram Bond were applied, as they are compatible with the coefficient of thermal expansion of the metal alloy used in this study (14.4×10^{-6}/k for the metal alloy), according to the manufacturer's instruction of the metal alloy used.

On the other hand, the mechanical bonding is the result of interlocking between the surface roughness of the metal and the ceramic layer [23]. In this study, through the roughness test on randomly selected strips from the main group A, it was observed that the metal surface was characterized by a large and irregular roughness, which led to unreadability of the surface roughness according to ISO standards adopted in the perthometer device used.

The irregularity of the metal surface manufactured by SLM technique is usually formed due to the adhered metal particles, in addition to the parameters of laser melting process, which also play a role in their formation [12]. While treating the metal surface by sandblasting with aluminum oxide particles, it led to reduction in roughness and production of a new and uniform roughness [12]. Therefore, there was a possibility in this study for obtaining Ra–Rz values for the specimens from the main group B treated with sandblasting. In addition, the sandblasting process improved the bond strength by increasing the ceramic wettability on the metal surface, as indicated in a previous study [24].

The three-point bending test was used in the A1 and B1 sub-groups, in which the bond strength exceeded the minimum required value of 25 MPa according to ISO 9693 standards, and this is consistent with a previous study [4]. Descriptive statistics demonstrated that the high roughness in the A1 sub-group did not allow for a full chemical bonding to be achieved as in the B1 sub-group, due to formation of voids and pores in the interspace, resulting from the high surface roughness that decreased the wettability of the porcelain, as indicated by previous studies [25, 26]. Also, Di *et al.* indicated that metal surface treated with grinding, involving natural surface roughness removal, and followed by sandblasting, would achieve a better bonding strength than sandblasting applied alone [27]. On the other hand, it was noted in other studies that there is no need for a relation between high roughness and increased bond strength [12, 23, 28-30].

Regarding bonding agent, through comparison between the A1 and A2 sub-groups, the application of the bonding agent had useful effect. On one hand, this can be explained by the fact that the application of bonding agent in the sub-group A2 contributed to an increase in ceramic wettability due to low viscosity properties of the bonding agent that led to a better bonding between the opaque layer and the metal. This contributed, in return, to a better metal-ceramic chemical bonding. This is consistent with previous studies which indicated that the application of the bonding agent led to the best porcelain wetting on the metal surface, while pores were formed in the interfacial region in specimens that were not subjected to the bonding agent, which confirms its' importance in increasing the porcelain wettability [25, 26].

Chen and Bonaccurso indicated that the wettability and liquid viscosity affect wetting dynamics, as the surface wettability increases when liquid viscosity decreases, and vice versa [31]. On the other hand, the surface structure with prominent roughness of the A2 sub-group specimens plays a role in improving mechanical interlocking [4]. According to the failure modes of comparison between the A1 and A2 sub-groups, we can assume that the presence of bonding agent significantly contributed to the improvement of bond strength in the A2 sub-group specimens.

In the comparison of the mean bond strength and fracture failure mode between the B1 with B2 sub-groups, there was no statistically significant difference since the application of the bonding agent in the B2 sub-group had a minor effect on improving the bonding strength compared with the B1 sub-group. This can be explained by the fact that the surface treated with sandblasting was characterized by a uniform and regular roughness that allowed for improving the metal-ceramic bond strength in the B1 and B2 sub-groups.

Overall, the test results showed that the mean bond strength of the studied sub-groups met the required bond strength according to ISO 9693 standards, which is 25 MPa, proving its' possibility for clinical application. Nevertheless, clinical studies are necessary to evaluate the long-term performance of SLM-based metal-ceramic prosthodontics.

CONCLUSIONS

Under the conditions of this study, it was found that the bonding strength in all the studied sub-groups exceeded the required minimum 25 MPa, according to ISO 9693 standards. The bonding agent had a minor effect on improving the bond strength between the ceramic and Co-Cr alloy when the metal surface was sandblasted. On the other hand, the bonding agent had an useful effect on improving the bond strength when the natural surface roughness from SLM was maintained; moreover, it resulted in a highest metal-ceramic bond strength value in the studied sample.

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CONFLICT OF INTEREST

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

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