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RESEARCH AND EDUCATION

The effect of layer thickness on the porcelain bond strength of laser-sintered metal frameworks

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ABSTRACT

Statement of problem. Laser sintering has become a common manufacturing technique in the fabrication of metal-ceramic restorations. The layer thickness of the sintering process may affect the surface morphology and hence the porcelain bond strength. However, limited information is available on the effect of layer thickness on porcelain bond strength.

Purpose. The purpose of this in vitro study was to evaluate the porcelain bond strength of direct metal laser-melted (DMLM) cobalt-chromium (Co-Cr) metal frameworks sintered with 25-µm and 50-µm layer thicknesses.

Material and methods. Thirty metal frameworks (n=10) were fabricated by using the lost-wax technique (group C [control]), DMLM with a 25-µm layer thickness (group L25), and DMLM with a 50-µm layer thickness (group L50) according to the International Organization for Standardization (ISO) 9693-1. The surface roughness of 1 metal specimen from each group was analyzed by atomic force microscopy. After porcelain firing, a 3-point bend test was applied to each metal-ceramic specimen as in ISO 9693-1. In addition, 1 metal framework from each group was prepared and examined by scanning electron microscopy to evaluate surface morphology. Data were analyzed statistically by using 1-way analysis of variance and the Tukey honestly significant difference tests (α=.05).

Results. Group C and group L25 showed significantly higher (P<.001) mean porcelain bond strength values than group L50, and no significant bond strength difference was found between groups C and L25. All groups generally exhibited an adhesive type of failure.

Conclusions. The results indicate that layer thickness may affect the porcelain bond strength of DMLM metal frameworks. (J Prosthet Dent 2019;122:76-81)

Metal-ceramic restorations are still one of the main prosthetic treatment options.[1,2] Fractures occurring at the metal-ceramic interface result in a time-consuming and costly problem for both clinicians and patients.[3,4] Therefore, an adequate bond strength between the porcelain layer and metal framework is essential for the long-term clinical success of metal-ceramic restorations.[4,5] The International Organization for Standardization (ISO) has specified that the minimum acceptable metal-ceramic bond strength value is 25 MPa.[6]

The bond in the metal-ceramic interface is primarily formed by the chemical interaction of the metal framework and ceramic opaque layer[7-11] and secondly by the micromechanical retention of these 2 layers.[7,8,10,11] Roughening the metal surface increases the wettability of the surface and enhances the metal-ceramic bond strength.[7,8,10] Therefore, the surface morphology of the metal framework plays a significant role in the success of metal-ceramic restorations.

Conventional lost-wax casting is a common fabrication method for metal-ceramic restorations.[12,13] Base metal alloys are frequently used in dental practice because of the high cost of noble metal alloys.[8,9,14] However, casting base metal alloys has complications such as their high melting range and susceptibility to oxide formation.[15,16] Unstable oxides weaken the metal-ceramic bond strength and eventually result in bond failure.[7,10] Moreover, sensitive casting procedures involve many time-consuming steps.[12,17] These disadvantages have been overcome by the introduction of computer-aided design and computer-aided manufacturing (CAD-CAM) systems.[18]

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Clinical Implications

Increasing the layer thickness decreases the manufacturing time but may weaken the porcelain bond strength of direct metal laser-melted cobalt-chromium frameworks.

CAD-CAM systems have evolved since their introduction, and current dental systems can be either subtractive (such as milling) or additive (such as laser sintering).[19,20] In recent years, the use of laser sintering systems in the fabrication of metal-based prostheses has increased because of the disadvantages of milling hard metal alloys.[17,19,20] Laser sintering devices used in dental applications generally work on the principle of direct metal laser sintering or direct metal laser melting (DMLM).[5] Both laser sintering systems use a laser source that consolidates metal powders layer by layer, thus transforming CAD data into a 3D structure.[12] Direct metal laser sintering devices partially melt the metal powders, whereas DMLM devices completely melt the metal powders.[5,21] Complete melting of the metal powder increases the structural density.[5,21]

Layering parameters affect the mechanical properties and structural details of laser-sintered metal structures.[22,23] Decreasing the layer thickness improves the mechanical properties. However, negative effects such as impaired surface finish, reduced accuracy, and decreased mechanical properties have been reported when the layer thickness exceeds a certain level.[5,24-29] The lowest and highest layer thickness values that can be selected are limited by the capacity of the laser sintering devices, and this range can vary between 20 μm and 100 μm.[5,22,30]

The effect of the layering parameters in the sintering process on the bond strength in the metal-ceramic interface is unclear. Therefore, the purpose of the present study was to compare the porcelain bond strength of cobalt-chromium (Co-Cr) frameworks fabricated by DMLM with 25-μm and 50-μm layer thicknesses. The null hypothesis was that no significant difference in bond strength would be found between the 2 layering parameters.

MATERIAL AND METHODS

The sample size of the present study was determined by performing a power analysis (G*Power 3.1.9.3. free software) based on preliminary tests to provide statistical difference (α=.05), and the effect size was found to be 8.98 at 99% power. A 3D bar-shaped metal framework (25×3×0.6 mm) was designed according to ISO 9693 by using a computer software program (Exocad; Exocad GmbH), and a total of 30 (n=10 in each group) Co-Cr alloy bars were fabricated (25×3×0.5 mm) by the lost-wax technique (group C), DMLM with a 25-μm layer thickness (group L25), and DMLM with a 50-μm layer thickness (group L50). The chemical composition and elastic moduli of the alloys are shown in Table 1.

Table 1. Chemical composition and elastic modulus of Co-Cr dental alloys

Groups	Elastic Modulus of Co-Cr Alloy (GPa)	Chemical Composition of Co-Cr Alloy (w %)
C	200*	59 (Co), 25 (Cr), 9.5 (W), 3.5 (Mo), 1 (Si), <1 (C, Fe, Mn, N)*
L25 and L50	195*	59 (Co), 25 (Cr), 9.5 (W), 3.5 (Mo), 1 (Si), <1 (C, Fe, Mn, N)*

C, lost-wax; Co-Cr, cobalt-chromium alloy; DMLM, direct metal laser melting; L25, DMLM with 25-μm layer thickness; L50, DMLM with 50-μm layer thickness. *Provided by manufacturer.

In group C, the CAD design was transferred to a 3D printing device (VIDA; EnvisionTEC GmbH), and 10 bar-shaped wax patterns (Press-E-Cast; EnvisionTEC GmbH) were prepared. The framework thickness was set at 0.6 mm to account for the finishing procedures. The wax patterns were invested with phosphate-bonded investment (Maruvest Speed; megadental GmbH) and cast in an induction-heated centrifugal casting machine (INF-2010; Mikrotek Dental) with a Co-Cr dental alloy (Starbond CoS; S&S Scheftner GmbH). After the separation of sprues, the cast specimens were airborne-particle abraded (APA) with 110-μm aluminum oxide (Al_2O_3) particles (Cobra; Renfert GmbH) at a pressure of 400 kPa and finished in one direction with tungsten carbide burs.

In groups L25 and L50, the CAD design was transferred to a DMLM machine (SLM-100; ReaLizer GmbH), and the framework thickness was set at 0.6 mm to account for the finishing procedures. In group L25, 10 bar-shaped metal frameworks were sintered with a 25-μm layer thickness from the Co-Cr dental alloy powder (Starbond CoS Powder 30; S&S Scheftner GmbH), and manufacturing was completed in 5.5 hours. In group L50, 10 bar-shaped metal frameworks were sintered with a 50-μm layer thickness from the Co-Cr dental alloy powder (Starbond CoS Powder 30; S&S Scheftner GmbH), and fabrication was completed in 2.5 hours. Then, all DMLM metal frameworks were subjected to an annealing procedure for 2.5 hours in a preheating furnace (Magma; Renfert GmbH) between 450 °C and 980 °C to relieve thermal stresses. After annealing, the DMLM specimens were separated from their supports, subjected to APA with 110-μm Al_2O_3 particles at a pressure of 400 kPa, and finished in one direction with tungsten carbide burs.

After the finishing procedures, the specimens were evaluated for an equal thickness of 0.5 mm by using digital calipers (Absolute Digimatic Caliper Series 551; Mituyoto). All the specimens were subjected to APA with 110-μm Al_2O_3 particles for 10 seconds at a pressure of 200 kPa and a distance of 10 mm at a 45-degree angle, steam cleaned (Triton SLA, Bego GmbH) for 5 seconds, and degreased with ethyl acetate for 10 seconds. Then, the surface roughness was analyzed (Fig. 1) for 1

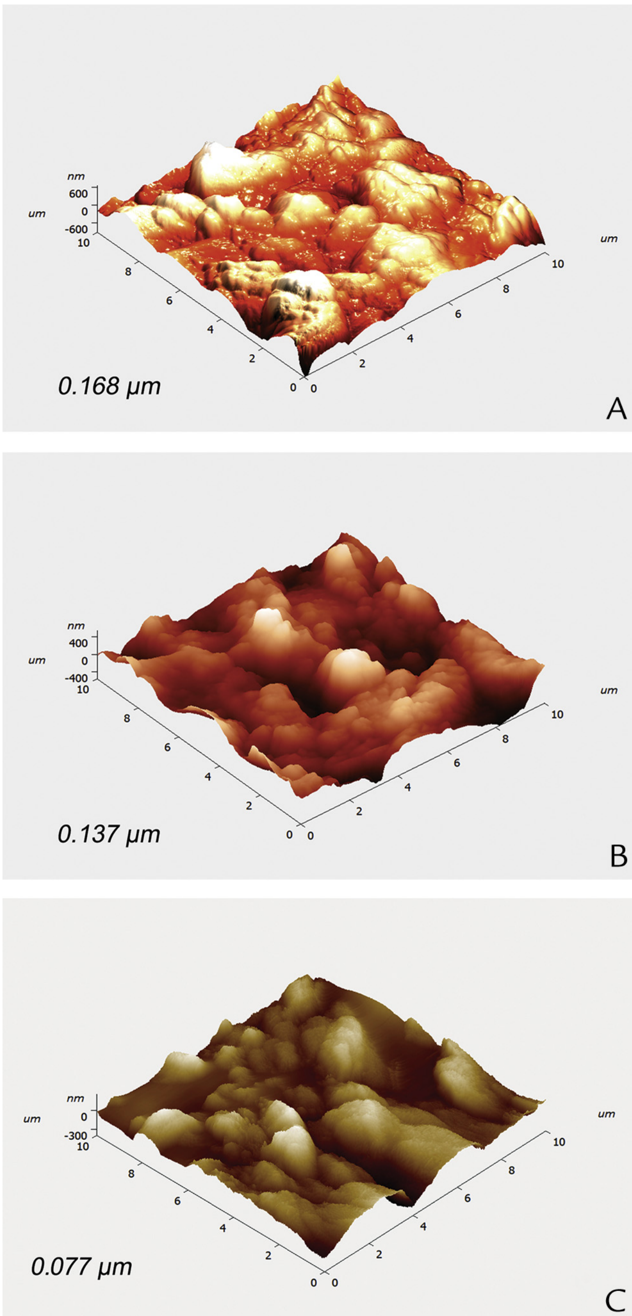

Figure 1. Surface roughness analysis by atomic force microscopy. A, Lost-wax. B, DMLM with 25-μm layer thickness. C, DMLM with 50-μm layer thickness. DMLM, direct metal laser melting.

specimen from each group by using atomic force microscopy (Solver Next; NT-MDT). Three measurements were recorded on each specimen with a sampling area of 100 μm, and the arithmetic mean of the 3 measurements was determined as the mean surface roughness (Ra) value of each specimen. All specimens were then oxidized according to the manufacturer's instructions, and a porcelain bonding agent (3C-Bond; Ceka Attachments Preci-Line), an opaque dentin and enamel porcelain (Vita VMK Master; Vita Zahnfabrik), and a glaze porcelain (Akzent Plus; Vita Zahnfabrik) were applied to the center section (8×3×1 mm) of all specimens by using a polytetrafluoroethylene mold. The specimens were fired (Programat P95; Ivoclar Vivadent AG) according to the manufacturer's instructions.

After porcelain firing, all specimens were stored in distilled water at 37 °C ±2 °C for 24 hours and thermocycled for 500 cycles between 5 °C ±2 °C and 55 °C ±2 °C with a dwell time of 30 seconds. A 3-point bend test was applied to all specimens by using a universal testing machine at a crosshead speed of 1 mm/min as specified in ISO standard 9693. The debonding strength was calculated by using the following equation: $\tau_b = k \times F_{fail}$, where τ_b is the debonding strength in MPa and F_{fail} represents the load in N at failure. The coefficient k is a function of the elastic moduli of the metal used, and its thickness is calculated by using the equation specified in ISO 9693-1. The type of failure was evaluated by using a stereomicroscope (SZX16; OLYMPUS) at a magnification of ×10.

In addition, 1 metal bar specimen from each group was prepared to evaluate surface morphology. Each specimen was examined under a scanning electron microscope (SEM; JSM-7001F; JEOL) after the annealing process (Fig. 2), after finishing procedures (Fig. 3), and after surface treatments (Fig. 4) to evaluate surface morphology.

Data were statistically analyzed by using a statistical software program (IBM SPSS Statistics, v21.0; IBM Corp). Differences among the groups were evaluated by using 1-way ANOVA and the Tukey honestly significant difference (HSD) tests (α=.05).

RESULTS

Results of the 1-way ANOVA test showed a significant bond strength difference (df=[2, 27], F=68.13, P<.001) among the groups. The lowest mean bond strength value was obtained from group L50 (Table 2). All specimens showed a higher bond strength value than 25 MPa. According to multiple comparisons, group C and group L25 showed significantly higher (P<.001) porcelain bond strength values than group L50, and no significant bond strength difference (P=.132) was found between groups C and L25. Adhesive failure was the most observed failure type in all groups (Table 3).

SEM images (Figs. 3 and 4) showed surface treatments altered the surface area but resulted in similar surface morphology. However, different roughness values were obtained from atomic force microscopy analysis. The Ra value was 0.17 μm for the cast specimen, 0.14 μm for the DMLM specimen with the 25-μm layer thickness, and 0.077 μm for the DMLM specimen with the 50-μm layer thickness.

DISCUSSION

The present study evaluated the porcelain bond strength of DMLM metal frameworks sintered with 2 different

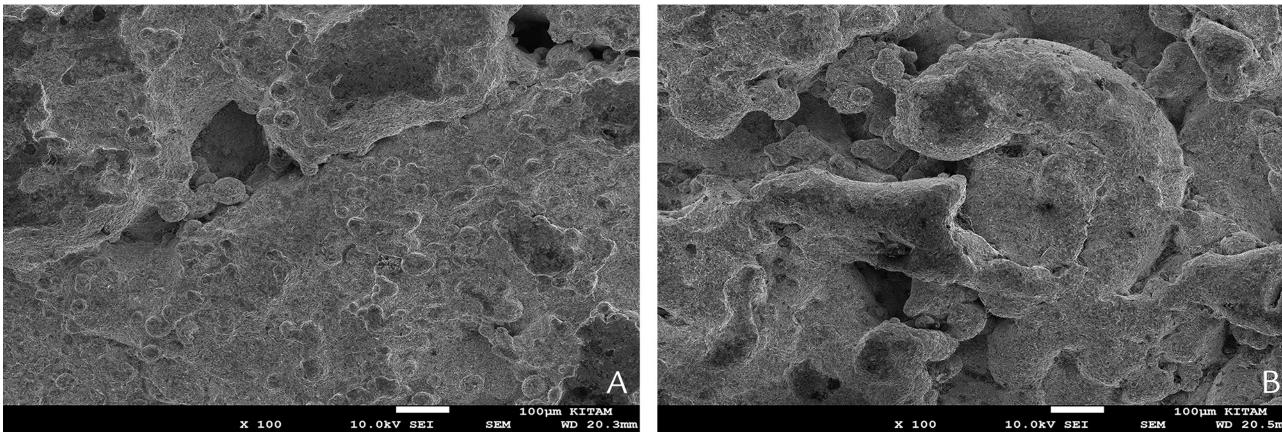

Figure 2. Scanning electron microscopy images after annealing process. A, DMLM with 25-µm layer thickness. B, DMLM with 50-µm layer thickness. DMLM, direct metal laser melting. Original magnification ×100.

layer thicknesses and revealed that the layer thickness affected the bond strength. Therefore, the null hypothesis that no significant difference in bond strength would be found between the 2 layering parameters was rejected.

The main bond formation between the metal framework and porcelain occurs chemically and involves ionic, covalent, and metallic bonds with oxides between the 2 layers.[7-11] The micromechanical interlocking of these layers also improves the bond strength. Roughening the metal framework alters the surface morphology and enhances micromechanical retention.[7,8,10] The surface morphology of the metal framework is influenced by the fabrication method used.[11,14,16]

DMLM devices work on the principle of powder bed fusion with selectable parameters determined by operators, such as layer thickness, which is the amount of powder distributed to melt on the surface.[29] Lower layer thickness means a higher manufacturing time.[29] Current devices used in dental applications work with a layer thickness of approximately 20 µm,[22] which is highly accurate.[22,23] However, layer thickness values of <20 µm may result in porosity within the structure. Moreover, high thickness levels that exceed a certain threshold may reduce accuracy and result in impaired surface quality.[5,24-29] This threshold value is not clear for dental applications.

Limited information is available on the effect of layer thickness on metal-ceramic bond strength. Ekren et al[5] reported no significant difference between the 20-µm and 30-µm layer thicknesses. Higher layer thickness values were tested in the present study. The specimens DMLM with a 50-µm layer thickness exhibited significantly lower ($P<.001$) bond strength values than the specimens DMLM with a 25-µm layer thickness. A thicker layer of powder is more difficult to melt due to insufficient penetration depth, which may lead to a balling effect within the structure.[31] A balling effect can be defined as porosity or delamination that causes poor interlayer bonding between the fresh powder and a previously sintered layer.[31] This phenomenon could be seen in the SEM image of the DMLM specimen with a 50-µm layer thickness recorded after the annealing process (Fig. 2). This may explain the low bond strength values obtained with a 50-µm layer thickness. Moreover, SEM images showed all specimens exhibited similar surface morphology after surface treatments. On the contrary, the surface roughness of the DMLM specimen with a 50-µm layer thickness was the lowest, and this might also have decreased the porcelain bond strength. Furthermore, although the 50-µm layer thickness group showed a higher porcelain bond strength value than 25 MPa, this difference may not be clinically relevant for long-term use.

No significant difference was found in bond strength (P=.132) between the control group and the DMLM group sintered with a 25-µm layer thickness. This may result from the similarity in elemental compositions. As stated before, the metal-ceramic bond is mainly chemical, and the metal ingots used in the casting process and the metal powder used in the laser-sintering process have the same elemental composition. Inconsistent with this finding, Wang et al[9] reported that the porcelain bond strength of laser-sintered specimens was significantly higher than that of cast specimens and attributed this result to the oxide layer. However, Akova et al,[14] Xiang et al,[3] and Bae et al[30] found no significant difference between the cast and laser sintering groups, consistent with the present study. Different test methodologies, laser sintering devices, and alloy types used in the studies may lead to different results.

The type of failure between the metal framework and porcelain is determined by evaluating the ceramic remnants after the bond strength test, and the failures are generally classified as adhesive, cohesive, or mixed.[5] In the present study, adhesive failure was mostly observed, and all adhesive failures occurred between the metal and metal oxide layers. Cohesive failures within the oxide layer are defined as Type V failures, which demonstrate a

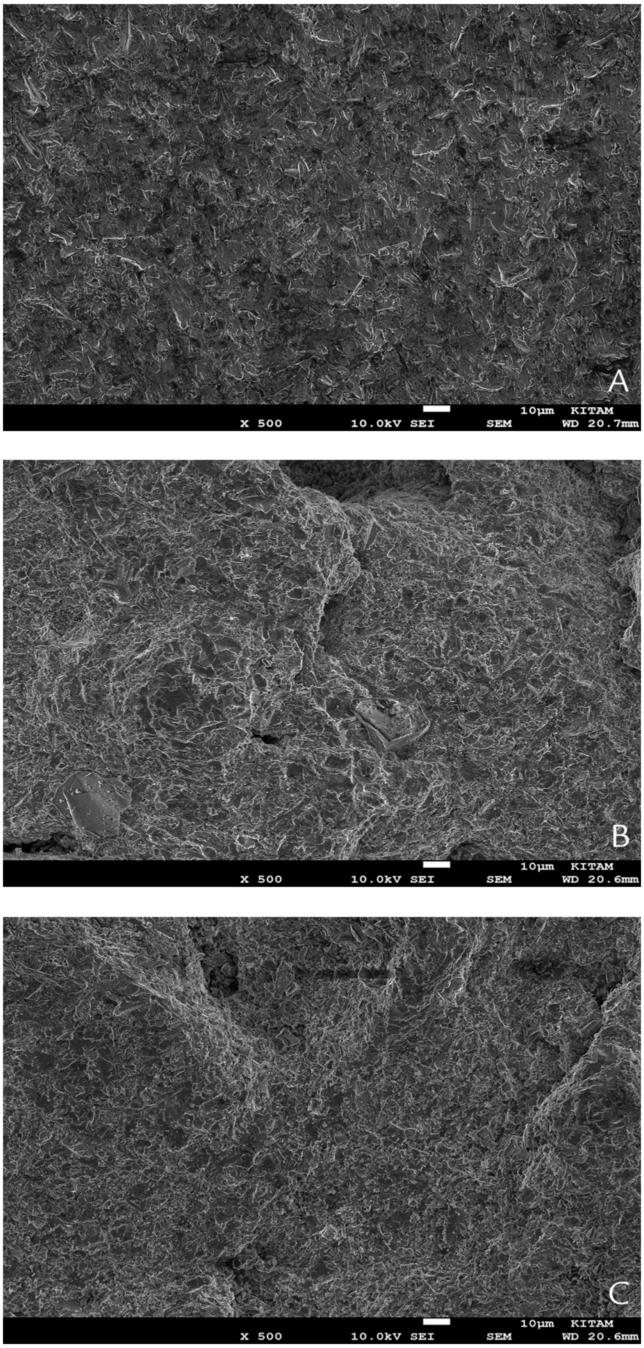

Figure 3. Scanning electron microscopy images after finishing procedures. A, Lost-wax. B, DMLM with 25-µm layer thickness. C, DMLM with 50-µm layer thickness. DMLM, direct metal laser melting. Original magnification ×500.

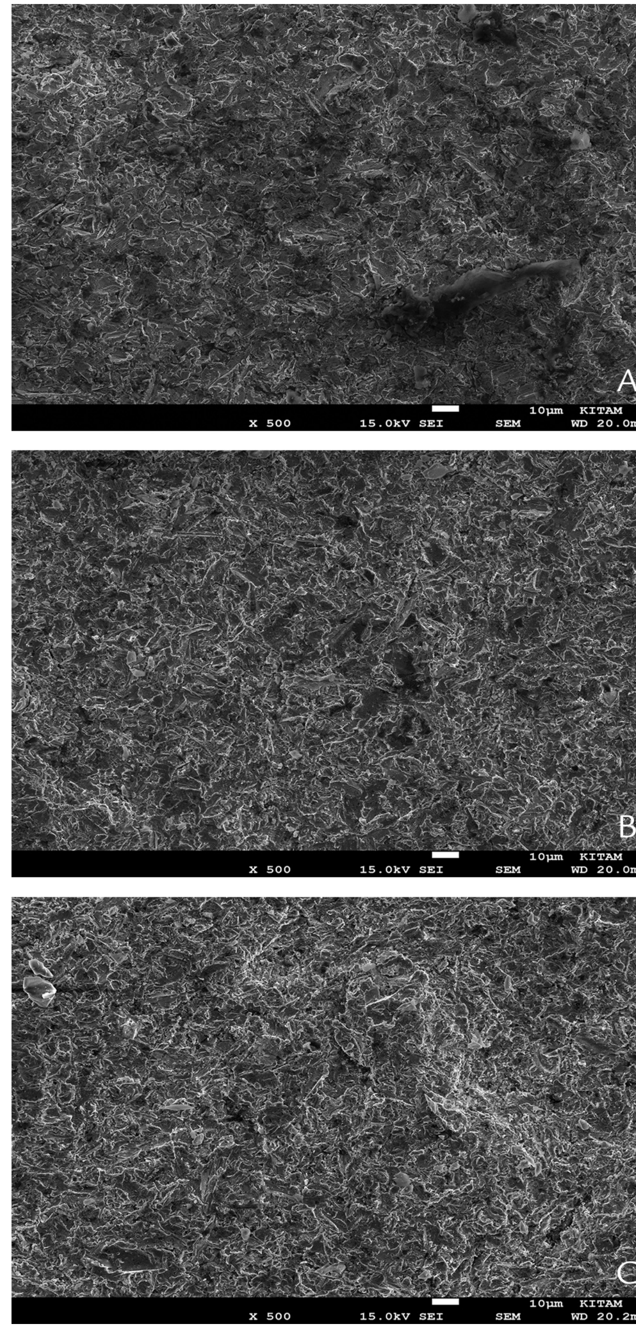

Figure 4. Scanning electron microscopy images after surface treatments. A, Lost-wax. B, DMLM with 25-µm layer thickness. C, DMLM with 50-µm layer thickness. Original magnification ×500. DMLM, direct metal laser melting.

weak porcelain bond strength.[32] No specimen exhibited a Type V failure in the present study.

Several test methods have been described to evaluate metal-ceramic bond strength; however, the results are variable due to different test geometries and stress distributions.[33-35] Shear tests have been reported to result in controversial results because of the nonuniform distribution of shear forces within the structure.[33] Various studies have used a 3-point bend test,[3,5,9,10] which is also recommended by ISO standard 9693-1 (2012).[6] Moreover, the 3-point bend test is a method that delivers reproducible numerical results.[36] Therefore, a 3-point bend test design was used in accordance with ISO 9693 in the present study.

A limitation of the present study was that only 1 type of laser sintering machine, dental alloy, and porcelain

Table 2. Mean ± standard deviation, minimum, and maximum bond strength values with statistical summaries

Groups	N	Mean (MPa)	±SD	Minimum	Maximum
C	10	47.75[a]	5.08	39.21	57.57
L25	10	51.06[a]	2.03	47.66	55.6
L50	10	32.91[b]	3.35	26.78	39.66

C, lost-wax; DMLM, direct metal laser melting; L25, DMLM with 25-μm layer thickness; L50, DMLM with 50-μm layer thickness; SD, standard deviation. No significant differences found between groups with same letter (P>.05).

Table 3. Type of failures

Groups	N	Adhesive	Cohesive	Mixed
C	10	8	–	2
L25	10	8	–	2
L50	10	10	–	–

C, lost-wax; DMLM, direct metal laser melting; L25, DMLM with 25-μm layer thickness; L50, DMLM with 50-μm layer thickness.

material was used to fabricate the metal frameworks. Also, the microstructural details and thickness of the oxide layer were not evaluated. Additional studies are required to reach more comprehensive results.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Layer thickness significantly affected (P<.001) the porcelain bond strength of metal frameworks, and the 25-μm layer thickness provided higher porcelain bond strength than the 50-μm layer thickness.
2. All specimens showed a higher bond strength value than the 25 MPa specified in ISO 9693.

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