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3D Printed Co-Cr Alloy Surface Treatments Effect on Shear Strength of Heat Cured Resins

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Abstract

Objective: To investigate the effect of different surface treatments—hydrochloric acid etching, sandblasting, metal primer application, and a combination of sandblasting with metal primer on the shear bond strength (SBS) of heat-cured polymethyl methacrylate (PMMA) bonded to 3D-printed Co-Cr alloy, and to assess the failure modes. **Materials and Methods:** Fifty disk-shaped 3D-printed Co-Cr specimens were divided into five groups (n=10 each): control (C), acid etch (A), sandblast (S), metal primer (P), and sandblast + primer (SP). Specimens were bonded to heat-cured PMMA resin and tested for shear bond strength using a universal testing machine. Failure modes were examined microscopically. Data were analyzed using one-way ANOVA and Duncan's multiple range test. **Results:** Significant differences in SBS were observed among groups ($p \leq 0.05$). The SP group showed the highest SBS (15.78 ± 1.19 MPa), followed by P (12.77 ± 2.61 MPa) and S (5.80 ± 1.01 MPa). The A (0.43 ± 0.08 MPa) and C (0.37 ± 0.07 MPa) groups exhibited the lowest SBS values. Failure mode analysis revealed adhesive failures in C, A, and S groups, while P and SP showed predominantly mixed failures. Sandblasting increased surface roughness, while primers enhanced chemical bonding without altering topography. **Conclusions:** The combination of sandblasting and metal primer achieved the highest bond strength, confirming a synergistic effect between micromechanical and chemical bonding. Acid etching alone was ineffective. Combined treatments are recommended to improve the clinical performance of 3D-printed Co-Cr RPD frameworks.

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Introduction

A removable partial denture (RPD) is an accepted device for treating patients with partial tooth loss. They help replace missing teeth and improve functionality. RPD usually consists of a base that supports the artificial teeth, along with a metal framework that provides structural integrity and stability. This combination ensures proper fit and function, enhancing the wearer's ability to chew and speak effectively. Cobalt-chromium (Co-Cr) alloy is often chosen for the framework due to its favorable properties, which include cost-effectiveness, elevated mechanical strength, excellent resistance to corrosion, and suitability for manufacturing processes [1,2].

Polymethyl methacrylate (PMMA) is commonly utilized as a denture base material for RPD due to tissue compatibility, aesthetic qualities, and favorable material characteristics [3,4].

Additive manufacturing (AM) technology, often referred to as three-dimensional (3D) printing technology, has increasingly been utilized to produce various dental prostheses [5,6]. With additive AM, we can create the metal framework using materials that are surprisingly like those traditionally used, like Co-Cr powder. This innovative approach opens up exciting possibilities for enhancing design and functionality [6,7].

Any flaws or separations occurring between the acrylic resin and the metal framework, especially along the finishing line, may lead to cracks or crazing in the acrylic resin. As a result, a weak bond can have a direct effect on the junction between the metal and the resin [4].

Traditionally, metal frameworks are secured to denture base resins using mechanical retention techniques, which can involve loops, meshes, beads, nail heads, and undercut finish lines.

Shear strength between the acrylic resin base and metal differs depending on the type of minor connector (latticework or mesh) [8].

The application of traditional mechanical retention methods for bonding metal to resin has

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been extensively investigated, yet it fails to eliminate microleakage [9].
Deficiencies in the chemical bond between metal and acrylic resin can lead to significant clinical issues, including microleakage of oral fluids. Such complications may be mitigated through the establishment of a robust chemical bond [10].
The chemical bonding of the denture base resin to the metal is preferred over mechanical retention in constructing RPD. Metal primers present a straightforward approach. They are a more economical choice relative to traditional methods. Special equipment is not necessary, and the application process is not particularly sensitive to technique [10,11].
This study was designed to evaluate the effect of various surface treatments on shear bond strength of heat polymerized acrylic resin bonded to 3D printed Co-Cr alloy.
The null hypothesis was that the shear bond strength between the Co-Cr alloy and 3D printed Co-Cr alloy would not differ, regardless of the types of various surface treatments.

Materials and Methods

Study design

In this study, 50 specimens were disk-shaped and used for the shear bond strength test. The specimens were divided into five groups based on the surface treatment performed on the specimens, each group had 10 specimens and as follows:
Group C: This is the control group in which no surface treatment was done on the bonding surface of the specimens.
Group A: The bonding surface of alloy samples in this group was treated with hydrochloric acid (10%).
Group S: The bonding surface of alloy samples in this group was sandblasted with110 µm aluminum oxide particles.
Group P: The bonding surface of alloy samples in this group was treated with metal primer.
Group SP: The bonding surface of alloy samples was sandblasted with 110 µm aluminum oxide particles, and then metal primer was applied.

Specimen Design

SketchUp (Trimble, USA) version 2024 was used to design the geometrical shape of the specimens. The specimens prepared for the assessment of shear bond strength were fabricated as disk-shaped specimens with a 10 mm diameter and 2 mm height [11-13]. All the designs were exported in an STL format.

Specimen preparation

The specimens were fabricated by a selective laser melting (SLM) machine (D-150, Riton, China), First, the STL files of the specimens' design were imported into the SLM software system and multiplied to the required number of specimens. All specimens were distributed virtually in software within the boundaries of the stainless-steel plate to be printed.
The samples were arranged on the platform so that their 3-mm wide sides were aligned with the z-axis, and the SLM was performed in a nitrogen environment with a laser energy of 10 kilowatts and an energy density of 1.964 J/mm^2.
The laser power (P) determined the energy density of 165W. The building direction was 90 degrees, the speed of the scan was 1050 (mm/s), and the scan line spacing was S. (mm). The particle size of the Co-Cr powder alloy (RITON, China) was between 12 and 65 microns, and the layer thickness was fixed to 25 microns [14].
The Co-Cr powder was applied to the stainless-steel plate. The machine started melting the powder layer by layer until the samples were fully constructed according to the manufacturer's guidelines.
When specimens' fabrication is finished, removing the support is easily done through the band saw (DLY-1BF1, Riton, China).
Finishing of the specimens was carried out with diamond grinding stones, then all the specimens were finished by using the abrasive wheel to remove any irregularities from the margin of the specimen [15,16].
For getting a good polished specimens surfaces a grinder polisher device (MP-160E,GOYOJO, Hong Kong) was used, the abrasive silicon carbide paper number 600 was used under running water for 10 seconds and the grinder speed was set to 300 rpm to provide a flat and uniform surface [11,12,17-19].

Application of Heat-Cured Acrylic Resin

To facilitate the fabrication of the acrylic wax pattern, a special split mold was made from stainless steel by a CNC machine to produce a wax pattern, a disk shape with 5 mm in diameter and 2 mm in thickness. The mold was fabricated to accommodate the Co-Cr disk with a dimension of 10mm in diameter and 2mm in thickness. Above the disk specimen, the mold would be narrowed in diameter up to 5 mm and extended 2 mm. This created a space of 5mm in diameter and 2mm in thickness in the center of the disk specimens, as displayed in Figure 1, where modeling wax (NK, China) was applied [11,12].
The final shape of specimen after application of molten wax is shown in Figure 2.
These specimens (Co/Cr alloy disk modeling wax assemblies) were flasked in a standard flasking technique for acrylic dentures with dental stone (Singletypo3,Lascod, Italy) and then the specimens were dewaxed and cleaned using boiled water [4].
Before the packing procedure, the specimens were divided into five groups, each with ten specimens, and then labelled according to the surface treatment they received.
For control group, the Co-Cr specimens without surface treatment.
For acid etch group, apply the hydrochloric acid 10% (Chemlab Hydrochloric Acid, AnalytiChem, Belgium) over the bonding surface of the Co-Cr specimens. After 30 minutes, the specimens were rinsed with deionized water for 30 seconds [20].
For air abrasive group, the specimens were abraded with aluminum oxide 110µm (Cobra Abrasive, Renfert, Germany) using a sandblasting machine (Sandblaster T1Mestra, Spain), at 4 bar pressure, and carried out for 14 seconds. The distance between the nozzle tip and the surface of the specimens was maintained at 2 cm, and held perpendicular to the tip [11].
For Metal Primer Group, 2- 3 drops of the metal primer (METAL PRIMER Z, GC; Japan) were dispensed into a clean dispensing dish, and then, using a brush, a thin layer was applied to the bonding surface and allowed to dry for 5 seconds. Immediately after that, the heat-cure acrylic was applied to the treated surface [11].
For specimens , to be treated with both sandblasting and metal primer, sandblasting was followed by application of metal primer [11].
After the surface treatment of samples, The heat cured acrylic resin(SR Triplex Hot, Ivoclar, Liechtenstein) mixed with powder: liquid ratio of 3:1 and packed by placing the flask in a hydraulic press (Flask Press, Quayle Dental, UK), with an applied pressure of 2000 psi, and then clamped for curing [21].
The clamped flasks were then placed in a water bath; the water was heated gradually from room temperature for about 45 minutes to a boiling temperature of 100C^o and maintained at this temperature for 30 minutes according to the manufacturer's instructions. The flask was allowed to bench cool slowly at room temperature [15].
The specimens were then deflasked and cleaned by using an ultrasonic cleaner(Electronic Scale) for 20 minutes, and then stored in distilled water(Al-Joud, Iraq) for 24 hours according to ADA specification number 12 [22].

Shear Bond Strength Test

The specimens inserted in the center of auto polymerizing acrylic resin base (Dentway Self-Cure, Dentway, Turkey) by using a special silicon mold. To obtain the bond strength. The specimens were mounted in the universal testing machine (HSA-UT, Dongguan Hongjin Test Instruments (DHTI), China) with the help of the specimen holder in a way that the treated specimen's surface was parallel to the loading piston, shown in Figure 3.
The loading piston had a chisel configuration, which was applied with a crosshead speed of

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0.5mm/min [11].The maximum load required at failure was recorded, and the shear bond strength was calculated by dividing the load by the surface area using the following equation: SBS (MPa) = F (N)/ S (mm^2).

SBS = shear bond strength (MPa), F = the load at failure (N), and S = surface area (mm^2). The load is the value at which the specimen deboned, and the surface area is the bonding area of the circular specimen that was 5 mm in diameter. The surface area was calculated according to the equation (s)=πr^2, where (r) represents the radius of the bonding area [23].

Following the shear bond strength test, the Types of failures were observed under an optical microscope (Hi-Scope; Hirox Co. Ltd.) at 10 X magnification. The mode of failure was categorized as adhesive failure if it took place at the interface between the resin and the metal, or cohesive failure if the separation occurred within either the metal or the resin. In instances where both types of failure were noted, the failure mode was classified as mixed-type failures [21,24,25].

Statistical Analysis

The Shapiro-Wilk normality test was first done. Mean, standard deviation, analysis of variance and Duncan's multiple rang test were carried out as statistical analysis for data using a statistical software program (SPSS version 25.0; IBM Corp, USA). The level of significance was set to α=0.05.

Results

All groups satisfied the normality assumption (all $P > 0.05$), and therefore one-way ANOVA was performed.

Table 1 shows descriptive statistics, including the mean values, standard deviation, standard error, and extreme values (minimum and maximum). The unit of measurement used is MPa.

Statistically, the lowest mean values were found in the Control group (0.369 ± 0.074 MPa) and then the Acid group (0.433 ± 0.081 MPa). The Sandblast group had a significantly higher mean value of (5.797 ± 1.011 MPa), while the Primer group had a statistically higher mean value of (12.769 ± 2.614 MPa). The combination group of sandblast and metal primer had the highest mean value (15.783 ± 1.193 MPa) (Table 1).

One-way ANOVA test was performed to determine if there was a significant difference between any of the studied groups. one-way ANOVA had a significance value of 0.000, so there was a significant difference between at least two groups, as shown in Table 2. Duncan's multiple range test was performed, which showed that there was no significant difference between only the Control and the Acid groups. However, there was a highly significant difference between all the other groups, as shown in Figure 4.

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Failure Mode Evaluation

The control (C), acid (A), and sandblast (S) groups all exhibited 100% adhesive failures, with no instances of cohesive or mixed failure observed. The metal Primer (P) group showed a clear shift in failure behavior: 60% of the samples exhibited mixed failures, while only 40% were adhesive. In the Combination group sandblast and metal Primer (SP), failure mode analysis revealed 70% mixed failures and only 30% adhesive failures, as showed in Table 3 and Figure 5.

Discussion

The findings of this study reveal significant differences in mean shear bond strength between 3D printed Co-Cr alloy and heat-cured acrylic resin when subjected to various surface treatments, such sandblast (S), metal primer (P), and sandblast +metal primer (SP), compared to the control group. However, there was no significant difference observed with the acid etch treatment in relation to the control.so, the null hypothesis was partially rejected.

The control group (C) and acid-etched group (A) exhibited the lowest shear bond strength values (0.369 ± 0.074 MPa and 0.433 ± 0.081 MPa, respectively), as showed in Table 1, indicating poor adhesion at the interface. These results suggest that acid etch or no surface treatment is insufficient for achieving clinically acceptable bond strength [26].

The sandblast group (S) showed a substantial increase in the shear bond strength (5.797 ± 1.011 MPa, as showed in Table 1, reflecting the positive effect of mechanical surface roughening. Sandblasting enhances micromechanical retention, which is well-documented as a mean to improve the bond between dental alloys and resin materials [25,27]. However, this group achieved significantly lower SBS values than the primer groups.

The primer group (P) and the combination sandblast +metal primer group (SP) achieved the highest shear bond strength values (12.769 ± 2.614 MPa and 15.783 ± 1.193 MPa, respectively) as showed in Table 1. The sandblast + metal primer (SP) group demonstrated statistically superior performance compared to all other groups. The synergistic effect of mechanical and chemical surface treatments is supported by literature, which showed that combining surface roughening with a suitable primer maximizes both micromechanical and chemical adhesion, resulting in a more durable and reliable bond [25,27].

The acid-etch surface group had lower shear bond strength values than the other surface treatments. This is maybe due to the presence of oxide film, which plays a vital role in the corrosion resistance of the Co-Cr material. Surface oxide film forms spontaneously on alloy surfaces after ambient oxygen exposure, acting as a barrier to electron flow (resistor) between the electrolyte and the alloy surface, thus protecting it against corrosion [28].

The oxide layer of the 3D-printed Cr-Co alloys has greater thickness and density than the cast alloys [29].

Furthermore, it is known that a harder material is capable of maintaining a thicker oxide layer more firmly as compared with a softer material [30], and it was found that the SLM-formed Co-Cr alloy has higher hardness than the cast alloy [14].

The result of this study agrees with previous work [31,32] that concluded that the use of HCL did not improve titanium–ceramic bonding strength values.

For aluminum oxide surface treatment group, the present study revealed that sandblasting significantly enhanced the shear bond strength (SBS) between the Co-Cr alloy and acrylic resin compared to the untreated control and acid-etched groups.

This finding aligns with several previous studies that confirmed the positive correlation between surface roughness and bond strength in metal-resin systems [20,33].

These findings are consistent with previous reports [20,34,35], which concluded that the Al_2O_3 abrasive significantly affects the quality of the Co-Cr alloy connection with the acrylic resin.

For metal primer surface treatment group, the findings of the current study revealed that the application of metal primer had a statistically significant positive effect on the shear bond strength (SBS) between the Co-Cr alloy and acrylic resin.

These results can be attributed to the chemical interaction facilitated by the functional monomers in the metal primer, most notably 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP). This monomer formed strong chemical bonds with the oxide layer of Co-Cr alloys, promoting adhesive interaction with the methacrylate groups in the resin matrix. This dual affinity enables the formation of a durable and stable hybrid layer at the alloy–resin interface, which significantly enhances SBS [36-40].

These findings are consistent with previous reports [13,20,41], which concluded that the metal primer significantly improved shear bond strength between acrylic resin and different metal alloys.

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Combination sandblast and metal primer surface treatment group (SP), the current study revealed that the combination treatment, resulted in the highest shear bond strength (15.783 ± 1.193 MPa) among all the experimental groups, as showd in Table 1. This remarkable increase in bond strength can be attributed to the synergistic effect of mechanical and chemical surface modifications [16].

These findings are consistent with previous reports [11,20,42], which concluded that the use of metal primers along with sandblasting significantly improved the bonding of acrylic denture base resin with the Co-Cr alloy.

However, this study disagrees with Ghazwan and Nabeel [43], who concluded that using metal primer combined with air abrasion improved the shear bond strength, but less than that obtained from specimens treated with metal primer only. This may be due to differences between the two studies, they used Valpast (flexible) denture base material and a different metal primer than that used in this study.

Limitations of this study included the *in vitro* design and the dimensions of the test sample used did not represent the actual clinical conditions. The difference in the geometry may affect the stress distribution and hence the shear bond strength and a single brand of metal primer was evaluated and there are additional surface treatments different from those used in this study that were not tested, like fiber laser and silica.

Conclusion

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

1. The combination of sandblasting and metal primer application demonstrated the highest shear bond strength, indicating a synergistic effect between micromechanical and chemical bonding. In contrast, untreated (control) and acid-etched groups showed low bond strength, emphasizing the limited impact of HCl alone.
2. All samples including: Control, sandblasted, and acid-treated groups exhibited 100% adhesive failures, indicating weak bonding, while the primer and SP groups displayed (60%-70%) mixed failures, confirming superior interfacial strength when chemical bonding is involved.

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Ethical Approval

Before starting this study, documented approval was obtained from the Research Ethics Committee (REC) at the College of Dentistry, University of Mosul, Iraq, with a license numbered (UoM.Dent/25/1011).

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None.

Conflict of Interest

None.

Data Availbility

Data are available upon reasonable request to the authors.

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Table 1. Descriptive statistics of the shear bond strength test for the studied groups.

Groups	N	Mean (MPa)	Standard Deviation	Minimum	Maximum
C	10	0.369	0.07415	0.27	0.46
A	10	0.433	0.08111	0.32	0.59
S	10	5.797	1.01101	4.43	7.43
p	10	12.769	2.61366	10.13	17.13
SP	10	15.783	1.19342	13.72	17.36

N: Total number of measurements. C: Control; A: Acid; S: Sandblast; P: Metal Primer; SP: Sandblast + Metal Primer.

Table 2. One-way ANOVA test for the shear bond strength test.

	Sum of Squares	Degrees of Freedom	Mean Square	F	p-value
Between Groups	1989.607	4	497.402	267.718	0.000
Within Groups	83.607	45	1.858		
Total	2073.214	49			

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Table 3. Distribution of failure modes between metal and denture base resin bonding following the shear bond strength test.

Group	Adhesive	Cohesive	Mixed
C	10	0	0
A	10	0	0
S	10	0	0
P	4	0	6
S P	3	0	7

C: Control; A: Acid; S: Sandblast; P: Metal Primer; SP: Sandblast + Metal Primer.

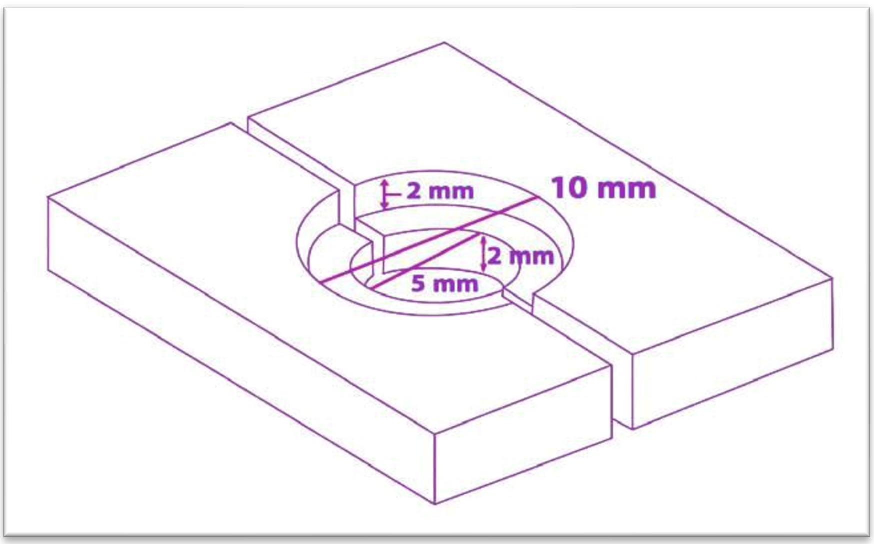

Figure 1. Diagram of the metal mold for wax application [11].

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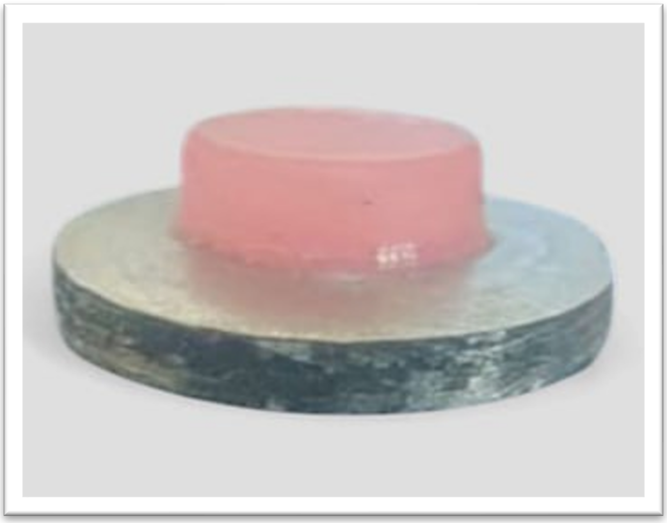

Figure 2. Final shear bond strength specimen shape after molten wax application.

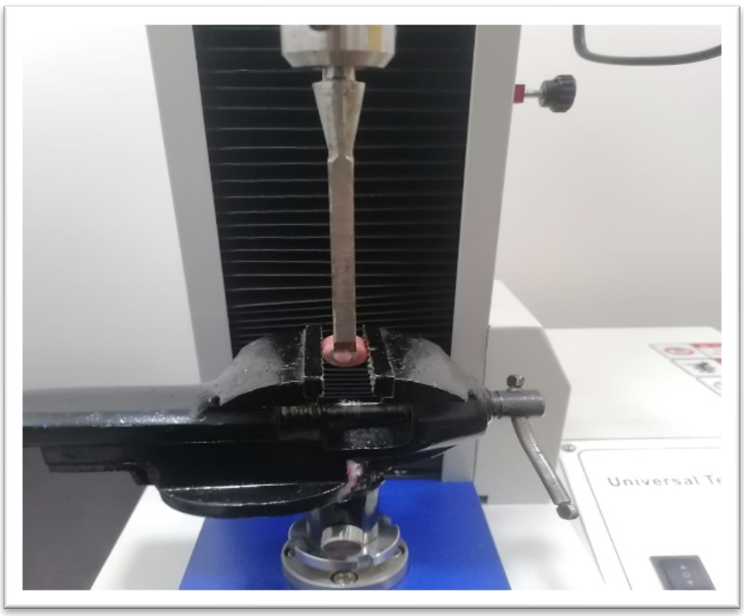

Figure 3. Specimens mounted in the universal testing machine.

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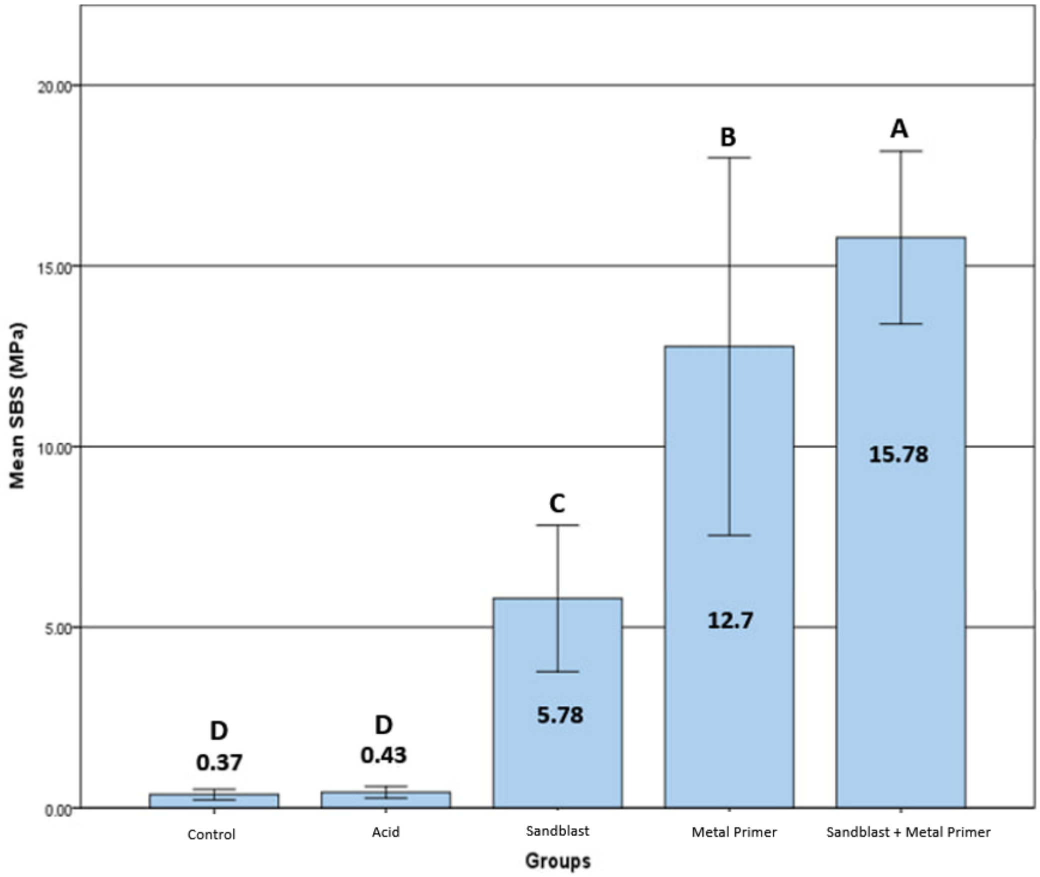

Figure 4. Duncan's multiple range test for the shear bond strength test for the studied groups.

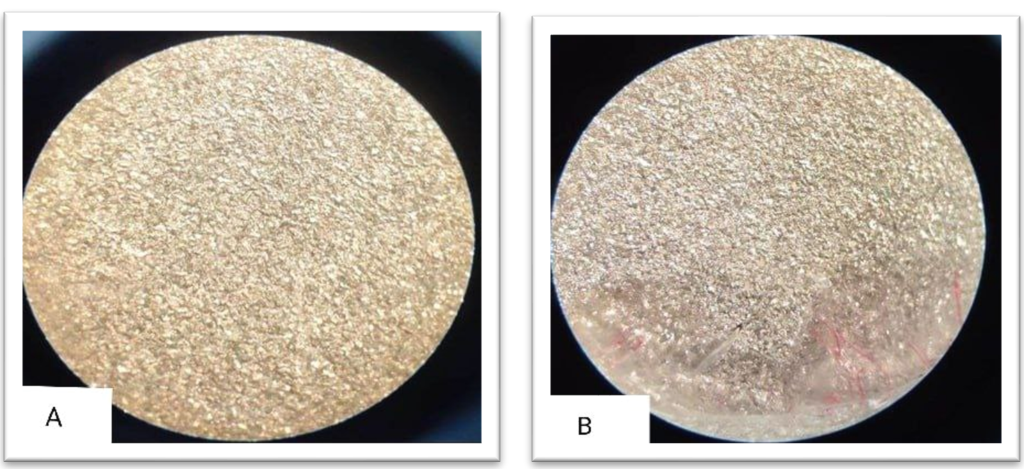

Figure 5. Types of failures: (A) adhesive failure. (B) mixed-type failure.