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Original Research

The Effect of Metal Primer on the Shear Bond Strength of Denture Base Resin to Base Metal Alloy after Electrolytic Etching and Laser Etching: An *In Vitro* Study

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Abstract:
Background: The purpose of this study was to compare and evaluate the effects of metal primer on the shear bond strength of denture base resin to base metal alloy after electrolytic and laser etching.
Materials and Methods: A total of 48 cobalt-chromium (Co-Cr) alloy specimens were fabricated. The specimens were divided into 6 groups of 8 specimens each. Group I: Sandblasting (control group). Group II: Sandblasting and metal primer application before the packing of acrylic resin. Group III: Electrolytic etching. Group IV: Electrolytic etching and metal primer application before the packing of acrylic resin. Group V: Laser etching. Group VI: Laser etching and metal primer application before the packing of acrylic resin. Each of these specimens was then subjected to shear load in Lloyd's universal testing machine.
Results: Laser etching with metal primer application showed the highest bond strength, followed by laser etching alone. Electrolytic etching without metal primer application showed the least bond strength.
Conclusion: Within the limitations of this *in vitro* study, the metal primer application after the metal surface treatments significantly increased the shear bond strength of denture base resin to Co-Cr alloy.

Key Words: Electrolytic etching, laser etching metal primer, sandblasting, shear bond strength

Introduction

Metal-resin bonding systems may be categorized as mechanical, chemical or a combination of the two.[1] Framework design must incorporate certain elements that provide mechanical retention of the resin.[2] The absence of a chemical bond directly affects the metal-resin interface. Microscopically, a space exists between the metal framework and the resin denture base. This contributes to increased microleakage of oral fluids, and microorganisms resulting in discoloration, foul odor, adverse soft tissue response, and deterioration of the denture base material.[3]

Recently, several chemical methods that enhance the bonding of resin to cast alloys have been developed, such as adhesive heat-cured opaque resin,[4] Silicoater system,[5] silane coupling agent and adhesive metal primer,[6] self-curing 4-methacryloxyethyl trimellitate anhydride or methylmethacrylate-tri-*n*-butylborane (MMA-TBB) opaque resin[7] and some commercially available adhesive bonding promoters. Kojima *et al*.[8] synthesized 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine 2,4-dithione (VBATDT) monomer and reported that an MMA-TBB resin bonded strongly to the precious metal with the use of VBATDT primer.

Barrack *et al*.[9] refined the metal framework by electrolytically etching the non-precious castings to improve the resin bond to the casting. Recently, laser surface treatment to enhance the micromechanical retention of bands and brackets has been introduced,[10] but the effect of this treatment on the bond strength of resin to non-precious alloy has not been investigated.

Hence, the purpose of this study was to examine the effect of metal primer on the shear bond strength of denture base acrylic resin to cobalt-chromium (Co-Cr) alloy after electrolytic etching and laser etching.

Materials and Methods

The materials used in this study have been listed in Table 1.

Preparation of alloy specimen (Figures 1a and b, and 2a and b)

Disk shaped specimens (10 mm in diameter and 2 mm in thickness) of base metal alloy were casted from the wax patterns using a standardized mold, sandblasted and divided into 6 groups of 8 specimens each.

Sandblasting was done using 150 µm alumina oxide particles, keeping the sandblasting point. 5 mm away from the surface to be etched for 60 s at a pressure of 0.15 MPa. The specimens were then cleaned in an ultrasonic cleaner for 10 min with distilled water. Each specimen was air dried and examined for bonding surface defects. Specimens with irregularities and defects were rejected. Specimens without defects were stored in individual plastic packets to avoid contamination until the surface treatments were done.

Surface treatments

Group I (Figure 3a)

This group consisted of metal specimens, which were only sandblasted. This group served as the control group.

Group II

This group consisted of sandblasted specimens on which the metal primer was applied later on.

Group III (Figure 3b)

This group consisted of sandblasted specimens, which were electrolytically etched. Equipment required for etching consisted of a variable low voltage direct current, rheostat, ammeter, and current meter. The metal specimens were attached to an insulated copper wire, which served as anode. A stainless steel electrode served as cathode. The electrolytic solution consisted of glycol and sulfuric acid. Cathode was placed into the solution maintaining a distance of 2 cm from anode. The specimens were etched in 250 ml solution for 6 min. A current density of 350 mA/cm^2 was maintained at an absolute density of 235 mA or 0.2 amps (current density × surface area). The specimens were then carefully removed from the unit avoiding contact with acid. Specimens were washed under running up water and then cleaned ultrasonically for 10 min. Then they were removed and washed in water for 2 min.

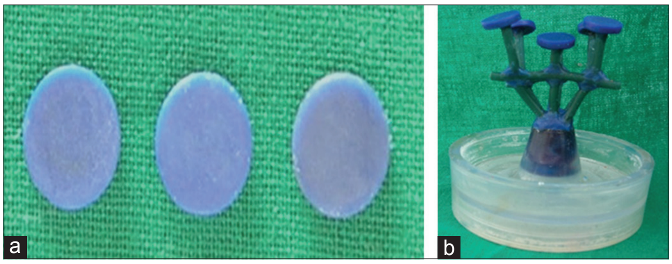


Figure 1: (a) Inlay wax patterns made out of silicon mold, (b) wax patterns sprued and attached to the crucible former.

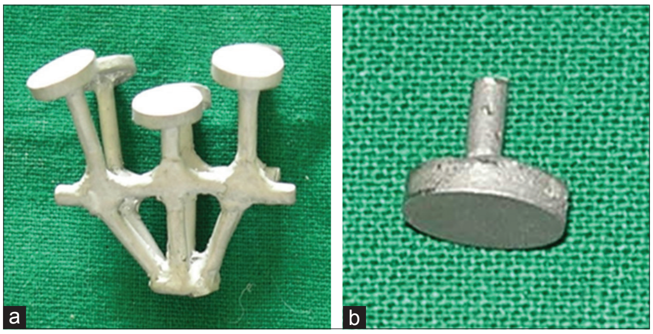


Figure 2: (a) Casting obtained after retrieval from the investment, (b) individual metal casting after cutting of the sprues and sandblasting.

Fresh solution was used each time as the solution tends to change the color slightly to a yellow hue. Samples were stored in plastic packets till they were removed to create wax patterns for the acrylic resin.

Group IV

This group was sandblasted and electrolytically etched by the procedure described above and on which metal primer was applied later on.

Group V (Figure 3c)

This group consisted of sandblasted specimens on which laser etching were carried out using Nd-YAG laser, which is basically used for industrial purposes.

Laser surface preparation was carried out using laser CHEVAL (Nd-YAG laser); CF 11-75(60). The CF 11-75 (60) is a high speed, high precision class IV laser micromachining system that uses coherent light energy to produce a mark on the surface of metals.

Laser etching technique

The metal disks were placed on the work surface behind the screen, which prevents the laser rays from affecting the eyes. The laser beam was focused on the metal disks.

The parameters used for laser surface treatments of the metal disks in the present study were; power 75 W, speed 1000 mm/s, frequency 8 KHz and a pulse length of 25 μs.

Group VI

This group consisted of sandblasted, and laser etched specimen on which metal primer will be applied later on.

Fabrication and bonding of denture base acrylic resin on to the casted metal specimen

Preparation of wax patterns over the metal casting (Figures 4 and 5a)

Wax patterns of 2 mm thick and 10 mm in diameter were obtained over the metal disks using PVC rings (10 mm in

Table 1: The materials used in this study.

Materials	Trade name	Type	Manufacturer	Batch no.
Co-Cr alloy	Co-Cr modelling legierung	Co-Cr pellets	Degussa	65743223S
Electrolytic etching solution	Wirolyt	H_2SO_4 and glycol liquid	BEGO, Germany	494
Metal adhesive primer	Alloy primer	VBATDT, MDP monomer	Kuraray Medical Inc.	241AD
Denture base resin	DPI heat cure improved	Heat cured resin	DPI India	764

VBATDT: 6-(4-vinylbenzyl-n-propyl) amino-1,3,5-triazine 2,4-dithione, Co-Cr: Cobalt-chromium

Figure 3: (a) Sandblasting of the metal casting, (b) electrolytic etching of Group III and IV, (c) laser etching of Group V and VI.

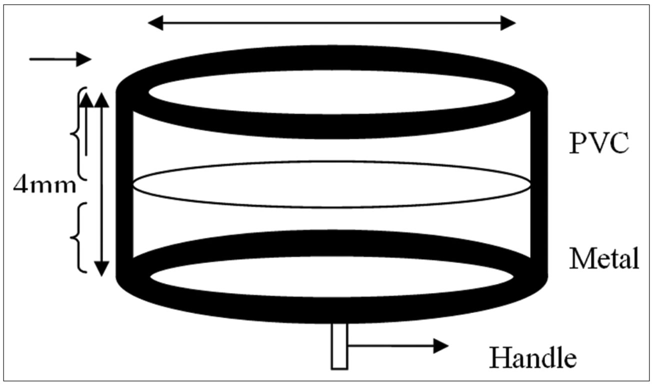


Figure 4: Diagrammatic representation of alignment of PVC ring over the metal casting.

internal diameter and 4 mm in length), which was aligned over the metal specimens.

Investing, dewaxing, and metal primer application

All the 48 specimens prepared for acrylic resin were flasked in a conventional denture flask and dewaxing was carried out.

The metal adhesive primer (alloy primer) was applied directly to the bonding surfaces of Group II, IV, and VI with a brush for 15 s and then air dried for 5 s.

Packing with heat-cure acrylic resin, curing, deflasking, trimming, and polishing (Figure 5b)

Heat cure resin was mixed according to manufacturers' directions, and the specimens were packed with three trial closures under 3000 Psi pressure and cured according to manufacturers' instructions.

The specimens were recovered, trimmed, and polished. Bonded specimens were stored in distilled water at 37°C for 24 h.

Testing of shear bond strength (Figure 5c)

Shear bond strength measurements were made using Lloyd's universal testing machine. The machine was operated at a crosshead speed of 1.0 mm/min and the maximum values to debond the specimen were recorded. The values thus obtained in Newtons were divided by the surface area to get the values in MPa. These values were then tabulated.

Results

Tables 2 shows the shear bond strength values of different groups. Analysis of variance, Scheffe's *post-hoc* test, independent-samples *t*-test and descriptive statistics were performed on the observed values (Table 2) for comparison between the groups.

Graph 1 shows the comparison of the mean shear bond strength of all the six groups.

The bond strength in increasing order was found to be as follows: Sandblasting and electrolytic etching < sandblasting alone < sandblasting with metal primer application < sandblasting and electrolytic etching with metal primer application < sandblasting and laser etching < sandblasting and laser etching with metal primer application.

Discussion

Results of this study indicated that control group (sandblasted only) in all the methods, recorded the least values of shear bond strength as compared to other groups except for the sandblasted and electrolytically etched group as seen in previous studies.[11] All the results were statistically significant. All the bond failures were adhesive indicating that the strength of acrylic resin is more than the bond strength between resin and Co-Cr alloy.[12]

Mechanical methods used in the present study to enhance the bond strength

Sandblasting

Sandblasting refers to the particle abrasion (alumina particles) of the alloy surface before bonding, to create irregularities and hence, a higher surface area substrate for binding, which has been shown to significantly improve the metal bonding.[13]

Electrolytic etching

Although electrolytic etching is currently considered to enhance the bond of resin to the surface of a metal casting, there are a number of disadvantages. For example, the surface of the cast metal retainer must be properly estimated to obtain the optimum current density. The essential requirement is that only those areas that are being etched should be exposed to the electrolyte. Setting proper time is also important, as longer time may lead to electropolishing of the surface and hence, deteriorate the bond. Another critical factor relates to the inter-electrode distance, which is normally 1.5-2 cm. As electrolyte itself acts as resistor, greater the electrode distance, greater the resistance.

Laser etching

Ablation by ultra-short laser pulses seems to be a very promising approach since this relatively new technology bears a high potential for a fast, precise and nearly damage-free material processing, also for broad industrial applications.

Different designs can be made on the metal surface using a laser, which is dependent on the operator's choice. Though the pattern seemed regular, there was no provision to measure the depth of the markings.

The pattern obtained by laser machining appeared to be quite regular with consistent depth unlike the uneven etching shown by sandblasting and electrolytic etching. This depth might be greater than that obtained by sandblasting or electrolytic etching, thus resulting in higher bond strength (Figure 6).

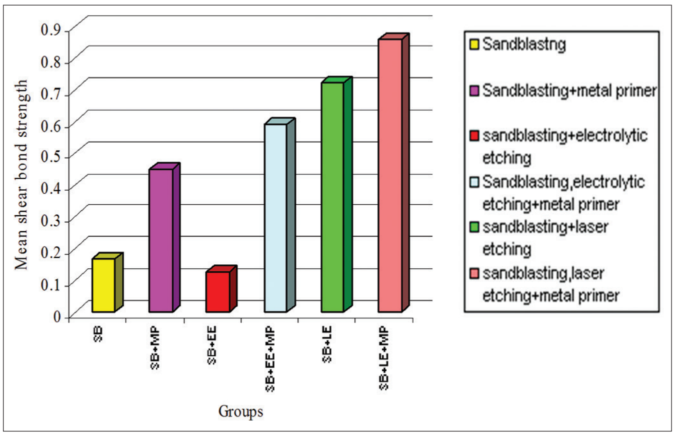
Chemical bonding to enhance the bond strength

A previous report stated that a strong adhesion mediated by a bonding agent may be free of any voids. Furthermore, the required thickness of the primer is less than the size of the bead or stud retention or any other physical means. Therefore, the bonding agent would preserve the necessary space for the artificial teeth or denture base resin and enhance the quality of the prosthesis.[14]

The result of this study indicated a positive influence of metal primer application after sandblasting, electrolytic etching, and laser etching, which significantly increased the shear bond strength of denture base resin to Co-Cr alloy. Surface irregularities

Table 2: Comparison of shear bond strength values of different groups.

Groups	*N*	Mean (Mpa)	Standard deviation
Group I	8	0.166428	1.4839
Group II	8	0.449695	2.0995
Group III	8	0.126750	9.3214
Group IV	8	0.591622	3.4105
Group V	8	0.721029	4.0381
Group VI	8	0.858009	2.6479
Total	48	0.485589	0.85800



Graph 1: Comparison of the mean shear bond strength of all the six groups, namely, sandblasting; sandblasting and metal primer application; sandblasting and electrolytic etching; sandblasting, electrolytic etching and metal primer application; sandblasting and laser etching; sandblasting, laser etching and metal primer application.

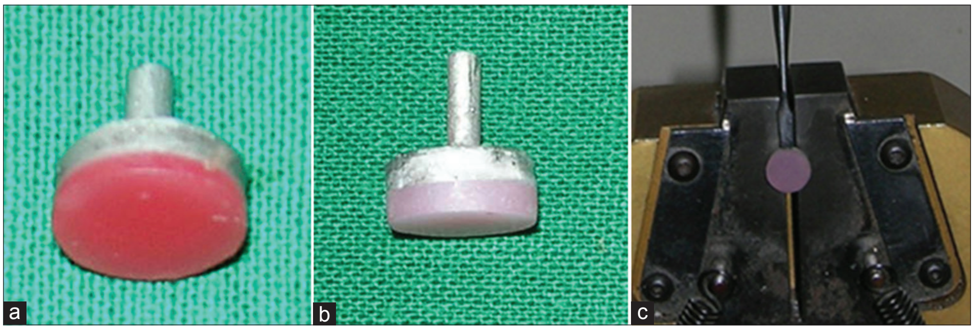


Figure 5: (a) Individual metal disc with wax pattern, (b) finished specimens, (c) shear bond testing using Lloyd's universal testing machine.

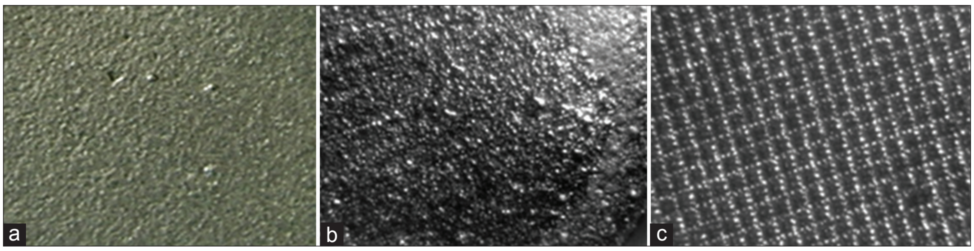


Figure 6: Metal surface under ×30 magnification, (a) sandblasting, (b) electrolytic etching, (c) laser etching.

as produced by sandblasting and their further wetting and removal of any air bubbles by metal primer application resulted in a better bond. Laser etching along with metal primer application enhanced the mechanical as well as the chemical retention thus giving the highest bond strength among all the groups.

Because the surface preparations for all the specimens for each were identical, the variation in this experiment was primarily affected by the nature of materials and by the investigator's ability to manipulate the materials.

Conclusion

Within the limitations of this *in vitro* study the following conclusions were made from the data obtained:

1. Sandblasting and electrolytic etching without the application of metal primer application is the least effective method
2. Laser etching the Co-Cr surface with metal primer application is the most effective method in increasing the shear bond strength of denture base resin to Co-Cr alloy.

Thus, it can be concluded from this study that metal primer application after the metal surface treatments such as sandblasting, electrolytic etching, and laser etching can significantly increase the shear bond strength of denture base resin to Co-Cr alloy.

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