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ORIGINAL ARTICLE

Evaluation of Shear Bond Strength of Composite Resin Bonded to Alloy Treated With Sandblasting and Electrolytic Etching

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Abstract Conservation of natural tooth structure precipitated the emergence of resin-retained fixed partial dentures. The weakest link in this modality is the bond between resin cement and alloy of the retainer. Various alloy surface treatment have been recommended to improve alloy–resin bond. This in vitro study was carried out to observe changes in the Nickel–Chromium alloy (Wiron 99, Bego) surface following sandblasting or electrolytic etching treatment by scanning electron microscope (SEM) and to evaluate the shear bond strength of a resin luting cement bonded to the surface treated alloy. 80 alloy blocks were cast and divided into four groups of 20 each. In groups-A & B, the test surfaces were treated by sandblasting with 50 and 250 µm sized aluminium oxide particles respectively. In groups-C & D, the test surfaces were first treated by sandblasting with 50 and 250 µm sized aluminium oxide particles respectively followed by electrolytic etching. Test surfaces were observed under SEM at 1,000× magnification. Two alloy blocks of each group were luted together by a resin luting cement (Rely X, 3M) and their shear bond strength was tested. The mean shear bond strength in MPa of groups-A to D were 6.44 (±0.74), 8.18 (±0.51), 14.45 (±0.59) and 17.43 (±1.20) respectively. Group-D showed bond strength that is more than clinically acceptable bond strength. It is recommended that before luting resin-retained fixed partial dentures, the fitting surface of the retainer should be electrolytically etched to achieve adequate micromechanical retention.

Keywords Resin-retained fixed partial denture · Electrolytic etching · Shear bond strength

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Introduction

In 1973, Rochette introduced the concept of bonding metal to teeth using flared perforations of the metal castings to provide mechanical retention for periodontal splinting [1]. His work suggested an alternative to the conventional fixed dental prostheses. These resin-retained fixed partial dentures replaced the missing dentition with minimum removal of tooth structure. The weakling in resin-retained fixed partial dentures was the weak bond between metal and resin rather than resin and enamel. Numerous methods have been designed to improve an adequate bond of composite resin-to metal. These approaches include micromechanical retention, macromechanical retention and chemical adhesion [2–5]. Commensurate with the improvements in metal bonding methods has been a broadened usage of resin-retained fixed partial dentures, also known as resin bonded prostheses.

Since its inception, electrolytic etching has been routinely used to enhance bond between metal and resin in resin-retained fixed partial dentures by mechanical retention methods. Economic factors restrict the use of expensive chemically bonding adhesive resin cements. Various surface treatments can be utilized to improve the mechanical bonding of resin-to base metal, e.g. sandblasting, electrolytic etching and chemical etching [4].

Not many studies have been carried out to assess the effects of various surface treatments of the Ni–Cr alloys on the bond strength with resin luting cements. This in vitro study was therefore undertaken to :

(1) Observe the qualitative surface changes in the Ni–Cr alloy surface following the sandblasting or electrolytic etching treatment by scanning electron microscope (SEM).
(2) Evaluate the shear bond strength of a commercially available resin luting cement bonded to Ni–Cr base metal alloy, surface treated by sandblasting and by sandblasting with electrolytic etching.

Materials and Methods

Following materials were used in this study-

(1) Nickel–chromium, beryllium free base metal alloy (Wiron 99, Bego, Germany)
(2) Investment material and liquid (Bellavast T and Begosol, Bego, Germany)
(3) Aluminium oxide particles (Korox, Bego, Germany), sizes 50 and 250 μm
(4) 0.5 N nitric acid
(5) Surface protection lacquer (Seculac, Bego, Germany)
(6) Dual-cure adhesive resin cement (Rely X, 3M, USA)
(7) Bonding agent (single bond, 3M, USA)
(8) Load of 2 kg with plunger

The study was carried out in the following steps-

Casting Sample Alloy Blocks

Eighty sample alloy blocks of dimensions 10 × 10 × 2.5 mm were cast using nickel–chromium, beryllium free base metal alloy (Wiron 99, Bego, Germany) by conventional method using phosphate bonded investment material and liquid (Bellavast T and Begosol, Bego, Germany) and an Induction casting machine (Fornax 35E, Bego, Germany).

Surface Treatments of the Cast Samples

All the 80 blocks were then randomly divided into 4 groups (groups-A, B, C & D) of 20 each and were subjected to four cycles of firing in porcelain furnace (Vacumat 100, Vita, Germany).

In group-A, the test surfaces to be bonded were treated with sandblasting by 50 μm aluminium oxide particles (Korox, Bego, Germany), at a distance of 10 mm, under 60 psi pressure, for 10 s by a sandblaster (Korostar, Bego, Germany), and then cleaned with steam cleaner (Triton, Bego, Germany) for 2 min. Sandblasting was indicated by a uniform matt appearance. In group-b, the test surfaces were sandblasted with aluminium oxide particles of 250 μm size by the similar procedure as described for group-A.

In group-C, the test surfaces were first sandblasted with 50 μm aluminium oxide particles in a similar fashion and were then electrolytically etched using 0.5 N nitric acid as an electrolyte. Each alloy block was attached with sticky wax to a 19 gauge stainless steel wire which was attached to the positive terminal of the current source. Thus each alloy block acted as the anode. The electrode wire and all the surfaces of the sample block, except for the surface to be treated and bonded, were covered with surface protection lacquer (Seculac, Bego, Germany) to protect them from the electrolytic action. Another 19 gauge stainless steel wire functioned as a cathode, at a distance of 1.5 cm from the anode in a glass beaker containing electrolyte. A current of 250 mA (current density 250 mA/cm^2) and 3 V DC was passed through the electrolyte for 5 min. A glass rod was used as a stirrer to stir the electrolyte solution, so that the evolving gas bubbles should not cling to the metal electrode surfaces and disrupt the current flow (Fig. 1).

The alloy block was then removed from the electrolyte solution and rinsed in cold running water. It was placed in a container with 18 % hydrochloric acid for 10 min in an ultrasonic cleaner (Ultraschall, Dentaurum) to remove the metal oxide layer. The alloy block was then held under cold running tap water to remove the acids. The surface protection lacquer was flaked off under running water. The samples were cleaned with a steam cleaner for 2 min. They were then air-dried and stored. Group-D samples were treated in the same manner as group-C samples, except that the group-D samples were sandblasted by 250 μm aluminium oxide particles prior to electrolytic etching. Surface appearance after different treatment of group samples is depicted in Fig. 2.

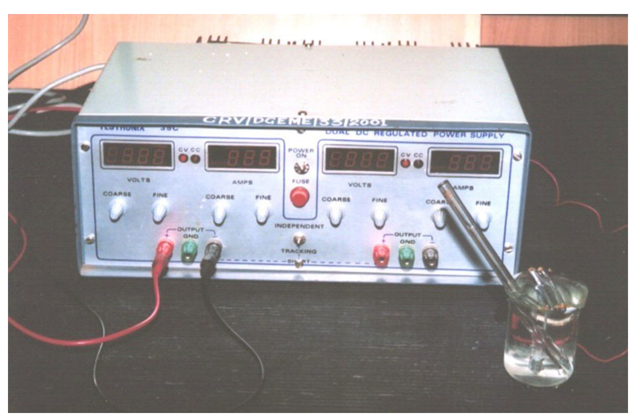

Fig. 1 Electrolytic etching being carried out

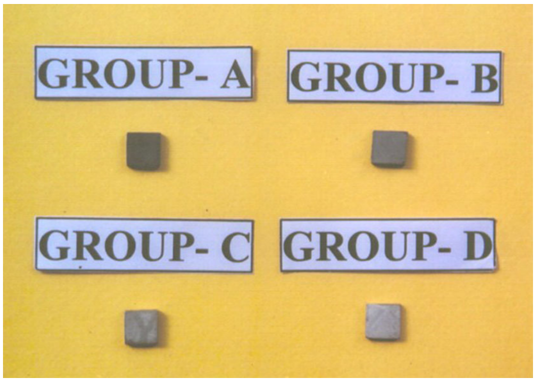

Fig. 2 Test surfaces after treatment

Observation by SEM

Test surfaces of all the alloy blocks, after they were surface treated, were observed under SEM (Kevex, Jeol) for qualitative surface appearance at 1,000× magnification.

Bonding Procedure

Two alloy blocks within each group were to be bonded to each other by a dual-cure adhesive resin cement (Rely X, 3M, USA). Prior to using resin cement, bonding agent (single bond, 3M, USA) was applied as per manufacturer's instructions on the test surfaces. For bonding, the cement was dispensed by a pre-measured dispenser (supplied by the manufacturer) and mixed for 10 s. It was then applied in a thin layer on the surfaces of the blocks and the two blocks were held together under a static load of 2 kg during cementation under the weight plunger. The excess cement was removed. The cement line at the interface of the two cemented blocks was light-cured for 40 s (as per the manufacturer's instructions) on all the four sides of the square-faced blocks. The load was released after the setting was complete, i.e. 10 min after light-curing. Two luted blocks constituted one sample, therefore, each group now had 10 samples.

Testing of Shear Bond Strength

The sample was tested on Instron Universal testing machine (Instron Corporation, Canton, Mass.) at a cross-head speed of 0.5 mm/min, as close to the cement interface as possible and shear bond strength was recorded.

Statistical Analysis

The results of shear bond strength testing were statistically analyzed by analysis of variance (ANOVA) and student's unpaired *t* test.

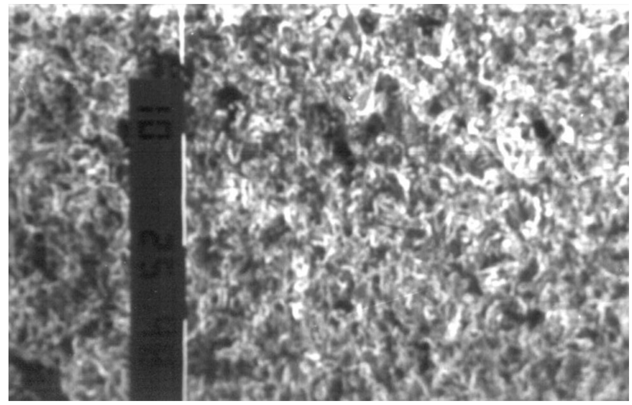

Fig. 3 SEM photomicrograph of group-A sample test surface after sandblasting with 50 μm alumina particles (×1,000)

Results

SEM Observations

Group-A

The samples showed pitted surface roughness but the surface irregularities were not marked (Fig. 3).

Group-B

The pitted surface roughness was more marked as compared to group-A as the blasting particles were five times larger in size (Fig. 4).

Group-C

The surface treatment had created linear and globulated 'screen-lattice' pattern of microstructural voids. The surface irregularities were not marked (Fig. 5).

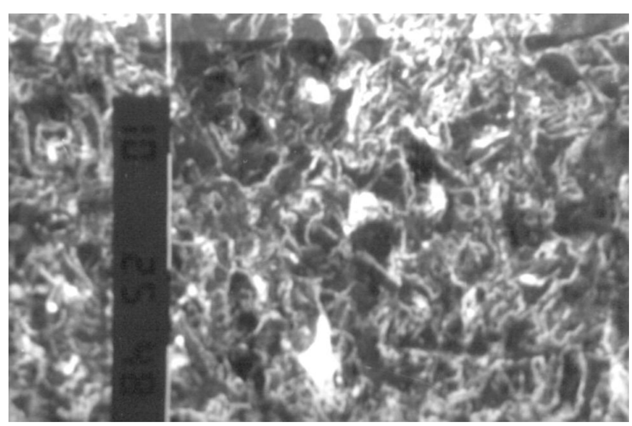

Fig. 4 SEM photomicrograph of group-B sample test surface after sandblasting with 250 μm alumina particles (×1,000)

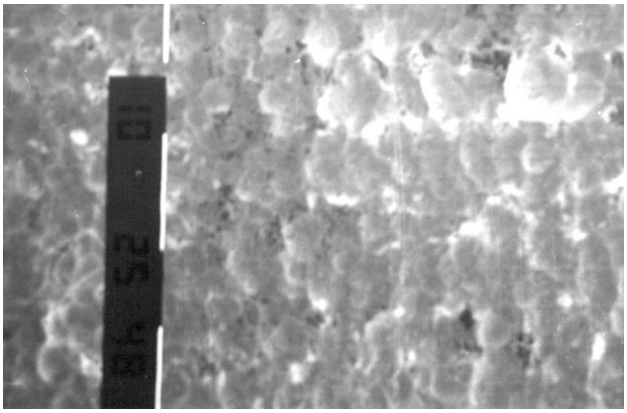

Fig. 5 SEM photomicrograph of group-C sample test surface after sandblasting with 50 μm alumina particles followed by electrolytic etching (×1,000)

Group-D

It showed marked surface irregularities (Fig. 6). It had deeper 'screen-lattice' pattern of microstructural voids and a wider, linear pattern of surface irregularities. The globulated appearance was marked and the linear depressions were wider and deeper as compared to group-C. This provided more of undulated areas.

Shear Bond Strength

The results of shear bond strength testing were tabulated. The mean values and standard deviations for each group were calculated (Table 1). Group-D recorded the maximum average shear bond strength, followed by groups-C, B & A, in decreasing order. The results were subjected to statistical analysis using ANOVA and it indicated that there was statistically significant difference in the average shear bond strength values between all the four groups, at $p < 0.001$ (Table 2).

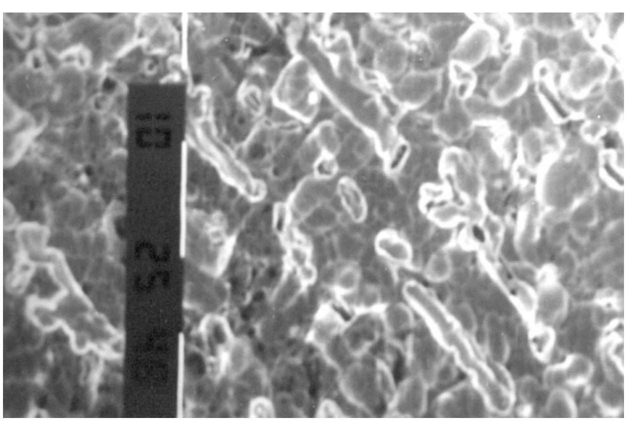

Fig. 6 SEM photomicrograph of group-D sample test surface after sandblasting with 250 μm alumina particles followed by electrolytic etching (×1,000)

Table 1 Statistical description

Parameter	Group-a	Group-b	Group-c	Group-d
Sample size (n)	10	10	10	10
Mean (x)	6.44	8.18	14.45	17.43
Range	5.23–7.46	7.27–8.78	13.76–15.24	15.86–19.17
Standard deviation [S.D. (±)]	±0.74	±0.51	±0.59	±1.20

Shear bond strength in MPa

Table 2 Analysis of variance (ANOVA)

Variation	Sum of squares (ss)	Degree of freedom (df)	Mean of squares (ms)	*f* value	*p* value
Between	805.00	3	268.34	411.42	$p < 0.001$
Within	23.48	36	0.652		
Total	828.48	39			

The calculated f value was greater than the table value of f at $p < 0.001$

Table 3 Statistical analysis to compare shear bond strength between different groups (at df = 18)

Compare groups	Calculated *t* value	Table value of *t*	*p* value	Significance of difference
A:B	4.8	3.92	$p < 0.001$	Significant
A:C	22.25	3.92	$p < 0.001$	Significant
A:D	3.52	3.92	$p < 0.001$	Significant
B:C	17.44	3.92	$p < 0.001$	Significant
B:D	25.72	3.92	$p < 0.001$	Significant
C:D	8.27	3.92	$p < 0.001$	Significant

All the calculated t values were greater than the table values of t at $p < 0.001$

To further analyze whether there was any significant difference in shear bond strength values between one group as compared to other three groups individually, a modified student's unpaired t test was carried out and the difference was statistically significant (Table 3).

Discussion

Since the advent of adhesive dentistry heralded by the introduction of acid-etch technique and composite resin, bonding technology has improved by leaps and bounds over the last 50 years. Conservation of tooth structure precipitated the emergence of resin-retained fixed partial dentures as a favoured alternative to the conventional fixed partial dentures [4]. A resin-retained fixed partial denture is a prosthesis that is luted to tooth structure, primarily enamel, which has been etched to provide mechanical retention for the resin cement. With the developments in adhesive techniques, the usage of resin-retained fixed partial denture also broadened. The weakling in these prostheses is usually the bond between metal alloy and adhesive resin.

Numerous methods have been developed to ensure an adequate bond of composite resin-to base metal alloys e.g.:

(a) Mechanical retention with perforations, as in Rochette bridges [1].
(b) Micromechanical retention by electrolytic etching, as in Maryland bridges [3]. They were able to achieve a resin-to-etched alloy bond that was stronger than resin-to-etched enamel bond.
(c) Macromechanical retention by Lost Salt Crystal method, as in Virginia bridges [5], but it had to have thick retainers.
(d) Chairside etching of fitting surface of the metallic retainer by using liquid (Assure-etch) or gel (Met-etch) chemical etchants containing Hydrofluoric acid [4] but the bond strength achieved was inferior to that achieved by electrolytic etching [6, 7].
(e) Chemical adhesion by Tin-plating, Silicoater system & Rocatec system [5, 8–10] but they require expensive and extensive equipments.
(f) Adhesion promoters, 4-methacryloxyethyl trimellitic anhydride (4-META) and tri-n-butyl borane (TBB) containing resin systems and 10-methacryloxydecyl dihydrogen phosphate (MDP) containing resin systems are capable of achieving a direct chemical bond between resin and base metal alloys [4, 5]. They are very costly & economic factor restricts their use.

Out of the various modalities of achieving retention of the resin with the metal surface, sandblasting and electrolytic etching enjoy more popularity because of economic viability.

With the new design concepts of resin-retained fixed partial dentures involving proximal grooves on the abutment teeth and 180° wrap-around effect, the component of tensile bond strength is not as much important as is the shear bond strength. Adequate shear bond strength is necessary to resist dislodging forces. Hence, the shear bond strength has been investigated in this study [5, 11–13].

Sandblasting causes surface roughness which leads to an increase in surface area and the numerous pits aid in micromechanical retention of the adhesive. Sandblasting with aluminium oxide particles of size 50 or 250 μm is the most commonly recommended surface treatment [4].

Electrolytic etching consists of selective anodic dissolution of certain metallic phases, thus forming microstructural voids that increase the surface area. This modified surface area offers better retention and enhances the bond strength. Electrolytic etching is an oxidation–reduction reaction [14]. It involves oxidation of metal into its anionic form. It requires an alloy with dendritic microstructure, such as base metal alloys, but it is not present in precious alloys. Hence, it is indicated for Ni–Cr or Co–Cr alloys. Here, the interdendritic eutectic phase is removed. The metal first becomes adsorbed on the surface and gets ionized. It then gets hydrated and precipitates into the solution. Ionized nitric acid is reduced to the gas, Nitrogen dioxide, which is dissipated into the air.

$$Ni \rightarrow Ni^{2+} + 2e^{-}$$

$$HNO_3 \rightarrow H^{+} + NO_3^{-}$$

$$H_2O \rightarrow H^{+} + OH^{-}$$

$$H_2O \rightarrow H^{+} + OH^{-}$$

$$Ni^{2+} + 2(OH^{-}) \rightarrow Ni(OH)_2\downarrow \text{ (ppt)}$$

$$NO_3^{-} + 2H^{+} + 2e^{-} \rightarrow NO_2 \uparrow \text{(g)} + H_2O$$

The surface area of the square sample alloy casting has been taken as 1 cm × 1 cm = 1 cm^2. which is clinically comparable to the surface area of most of the resin-retained fixed partial denture retainers. It also simplifies the result calculation [6, 15].

The samples were sandblasted for 10 s, which is a sufficient time to achieve surface roughness for micromechanical bonding and with effective, practical and safe distance and pressure [10, 16–18].

The electrolyte used for electrolytic etching was 0.5 N nitric acid. It is the most favoured electrolyte for Beryllium free Ni–Cr alloy [19–21]. Inter-electrode distance of 1.5 cm, voltage at 3 V DC, current density of 250 mA/cm^2 and 5 min' time for electrolysis are recommended parameters for electrolytic etching of Ni–Cr alloy [15].

For bonding of samples, it has been suggested that to improve the 'wetting' of the treated alloy surface by the resin, unfilled resin or resin bonding agent should be applied on the alloy surface first and then resin cement should be applied [6, 7, 20, 22–24]. During bonding, a constant load should be applied for the purpose of standardization [25]. A plunger weight of 2 kg/cm^2 of surface area being luted is adequate [20]. There is no significant difference in the bond strength whether the samples were thermocycled or not [26].

Because of its size and higher momentum, when a larger particle (250 μm alumina) hits the alloy surface during sandblasting, it creates five times larger pit-like depression as compared to a smaller particle (50 μm alumina), which is supported by SEM observations. When adhesive resin cement is applied to the alloy surface, the resin easily flows in the larger pits and forms a larger and stronger resin tag inside them, which accounts for its enhanced micromechanical bond. Electrolytic etching creates surface roughness *further* to that created by sandblasting, by increasing the surface area and hence, increased bond strength by micromechanical retention. Hence, following sandblasting with 250 μm alumina particles, a greater surface area is available for electrolytic etching, leading to greater surface irregularities and voids. The globulated appearance is marked and the linear depressions are wider and deeper as compared to group-C, as observed under SEM, which accounts for greater micromechanical bond strength.

The mean shear bond strength achieved by sandblasting alone is lesser than the clinically acceptable resin-to-enamel bond strength but shear bond strength achieved in this study by sandblasting followed by electrolytic etching is higher than the clinically acceptable resin-to-enamel bond strength (8.5–9.9 MPa) [6].

It is recommended that further research and clinical trials using different commercial products of adhesive resin cements and alloy must be carried out to substantiate the data base.

Summary and Conclusion

The results of this study led to the following conclusions:

(1) Sandblasting of the test surfaces produced pitted surface roughness, which was less marked with 50 μm alumina particles and more marked with 250 μm alumina particles.
(2) The shear bond strength achieved by only sandblasting the test surface of the alloy is lesser than the clinically acceptable enamel-to-resin bond; therefore, surface treatment by only sandblasting may not be adequate for clinical practice.
(3) Sandblasting with 250 μm alumina particles followed by electrolytic etching of the test surfaces resulted in maximum shear bond strength.
(4) It is suggested that before luting resin-retained fixed partial dentures, the fitting surface of the retainer should be electrolytically etched to achieve adequate micromechanical retention.

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