

Bond strength of resin composite to differently conditioned amalgam

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Bulk fracture of teeth, where a part of the amalgam restoration and/or the cusp is fractured, is a common clinical problem. The aim of this study was to evaluate the effect of different surface conditioning methods on the shear bond strength of a hybrid resin composite to fresh amalgam. Amalgams ($N = 84$) were condensed into acrylic and randomly assigned to one of the following treatments ($N = 6$): (1) Alloy primer + opaquer, (2) Air-particle abrasion ($50 \mu\text{m Al}_2\text{O}_3$) + alloy primer + opaquer, (3) Silica coating ($30 \mu\text{m SiO}_x$) + silanization + opaquer, (4) Opaquer + pre-impregnated continuous bidirectional E-glass fibre sheets, (5) Silica coating + silanization + fibre sheets, (6) Silica coating + silanization + opaquer + fibre sheet application. Non-conditioned amalgam surfaces were considered as control group (7). The mean surface roughness depth (R_z) was measured from the control group and air-abraded amalgam surfaces. The resin composite was bonded to the conditioned amalgam specimens using polyethylene molds. All specimens were tested under dry and thermocycled ($6.000, 5-55^\circ\text{C}, 30 \text{ s}$) conditions. The shear bond strength of resin composite to amalgam substrates was measured in a universal testing machine (1 mm/min). Surface roughness values for the non-conditioned control group ($R_z \sim 0.14 \mu\text{m}$) and for air-particle abraded surfaces with either Al_2O_3 or SiO_x ($R_z \sim 0.19 \mu\text{m}$ and $R_z \sim 0.16 \mu\text{m}$, respectively) did not show significant differences ($p = 0.23$) (One-way ANOVA). In dry conditions, silica coating and silanization followed by fibre sheet application exhibited significantly higher results ($14.8 \pm 5.6 \text{ MPa}$) than those of the groups conditioned with alloy primer ($2.2 \pm 0.7 \text{ MPa}$) ($p < 0.001$), air-particle abrasion+alloy primer ($4.4 \pm 2.0 \text{ MPa}$, $p < 0.001$), silica coating + silanization alone ($6.2 \pm 0.8 \text{ MPa}$, $p = 0.009$) or non-conditioned group (1.4 ± 0.6 , $p < 0.001$). Silica coating and silanization followed by additional fibre sheets with opaquer application ($23.6 \pm 6.9 \text{ MPa}$) increased the bond strength significantly compared to those of other groups (group 5 vs group 6, $p = 0.007$; other groups vs group 6, $p < 0.001$). Thermocycling decreased the bond strengths significantly for all of the conditioning methods tested (for group 1, $p < 0.001$; for group 2, $p = 0.013$; for group 3, $p = 0.002$; for group 4, $p = 0.026$; for group 5, $p = 0.002$; for group 6, $p < 0.001$ and for group 7, $p < 0.001$).
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Clinical relevance

Combination of silica coating and silanization with addition of E-glass fiber sheets at the adhesive interface can be considered as an alternative method to improve adhesion of resin composite to amalgam.

Introduction

Amalgam has served dentistry for more than a century. Although amalgam fillings undergo constant corrosion and they might not fulfil all cosmetic-esthetic demands, they are still commonly used. The results of recent

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surveys from cross-sectional studies indicate that complete cusp fracture of posterior teeth associated with amalgam restorations is a common problem in dental practice. The failure rate range between 4.4 [1] and 14 occasions [2] per 100 subjects or 20.5 teeth per 1000 persons a year [3].

A number of factors seem to contribute to the fracture of teeth with or without loss of tooth substance and amalgam material, such as occlusal instability, impact load, fatigue load during mastication, secondary caries, microdefects, technical errors, insufficient sound tooth material available surrounding the restoration (undermining cusps) or occlusal prematurity. In addition, the more surfaces restored and/or the wider the isthmus, the greater the chance of cusp fracture [4–7]. Thus it is likely that the restorative status of the tooth has an influence on the incidence of fracture. The majority of the fractures were observed in the supragingival location, which suggested that the fractured tooth could be restored easily [3]. Although there is little published literature on the subject, repair of a restoration is more cost-effective than total replacement where ever appropriate [8, 9]. It can be considered as fitting in a trend towards a less interventionist procedure [10].

Recently, veneering the restorations with resin materials has been attempted to solve the problem of repairing fractured teeth with amalgam restorations. Various repair techniques have been suggested in the literature, many of which are based on either mechanical and/or chemical adhesion techniques [11–17]. Mechanical means include roughening the amalgam, preparing undercuts, creating grooves or placing self-threading pins [18]. Chemical means on the other hand, use multipurpose adhesive agents [19, 20]. While primers and adhesives form ionic bonds with metal oxides or with the active metal compounds of the amalgam [21], several studies have shown that air-borne particle abrasion modifies the metal surface and provides micro-roughness that is essential for mechanical bonding [12, 15, 20]. However, the existence of a true chemical bond between amalgam and resin composites is controversial [14, 22].

The techniques that facilitate alloy-resin bonding have significantly improved over the last decade [23] and rely on both (micro)mechanical and chemical adhesion. Numerous intraoral repair systems are available and a growing number of systems are being introduced. Modern surface treatment methods mostly require air-borne particle abrasion of the metal prior to bonding. These new systems also involve the conditioning of the substrate to produce bifunctional silane molecules that adhere to the metal surface after being hydrolyzed to silanol and forming polysiloxane network on the substrate and finally reacting with the monomers of the opaquer/composite [24]. One system, in which silanes are also used, is tribochemical silica coating. The surfaces are air-abraded with 30 μm grain size aluminium

trioxide modified with silicic acid. The blasting pressure results in embedding of silica particles on the surface rendering the surface chemically more reactive to resin via silane. One other repair alternative has been proposed with the use of reinforcing fibers for the composite where improved fatigue resistance of composites were noted [25].

The literature contains many reports on bonding of resin composites to alloy surfaces but these methods have not been investigated for the purposes of bonding composites to amalgam. Although the concept of veneering amalgam restorations with composite is not new to restorative dentistry, there seems to be no consensus in the literature regarding the best method for repairing such restorations. Therefore the aim of this study was to assess the bond strength between amalgam and resin composite mediated by recent surface conditioning methods.

Methods and materials

The amalgam (non-gamma 2, lathe-cut, high-copper alloy with 43% Ag, 25.4% Cu) ($N = 84$) (ANA 2000 Duet, Nordiska Dental AB, Angelholm, Sweden) was triturated according to the manufacturer's recommendations regarding speed and time and then condensed with a hand instrument into a cylindrical (diameter: 6 mm, depth: 2 mm) undercut cavity prepared in autopolymerized PMMA (Palapress, Vario, Heraeus Kulzer, Wehrheim, Germany) until they were slightly overfilled. The exposed surface of each specimen was ground finished to 1200 grit silicone carbide abrasive (Struers RotoPol 11, Struers A/S, Rodovre, Denmark) and cleaned for 10 minutes in an ultrasonic bath (Quantrex 90 WT, L&R Manufacturing Inc., Kearny, NJ 07032-0607) containing distilled water and air-dried. Subsequently, the amalgam specimens ($n = 6/\text{group}$) were randomly distributed in seven testing groups according to their surface treatment (Table I and Fig. 1):

Surface conditioning methods

Group 1: In the alloy primer treated groups, the primer [6-(4-vinylbenzyl-n-propyl) amino-1,3,5 triazone-2,4-dithiol(VBATDT)] (Alloy PrimerTM, Kuraray Medical Co., Ltd, Tokyo, Japan) was first applied on amalgam substrates and it was allowed to dry prior to bonding procedures.

Group 2: Alloy primer was applied following air-borne particle abrasion with 50 μm Al_2O_3 (Korox[®], Bego, Bremen, Germany) using an intraoral air abrasion device (Dento-PrepTM, RØNVIG A/S, Daugaard, Denmark) from a distance of approximately 10 mm at a pressure of 2.5 bars for 4 s.

Group 3: Silica coating process was achieved using the same device under the same conditions but this time it was filled with 30 μm SiO_x (CoJet[®]-Sand, 3M

TABLE I Surface treatment and testing groups ($n = 6$ /per group)

Conditioning principle	Group 1	Group 2	Group 3	Group 4	Group 5	Group 6	Group 7
Air particle abrasion (50 μm aluminium trioxide)		✓					
Silica coating (CoJet [®] -Sand, 30 μm SiO _x)			✓		✓	✓	
Alloy primer	✓	✓					
Silane coupling agent			✓		✓	✓	
Pre-impregnated bidirectional E-glass fibre sheets				✓	✓	✓	
Opaquer	✓	✓	✓	✓		✓	
Bonding agent					✓	✓	
Composite resin	✓	✓	✓	✓	✓	✓	✓

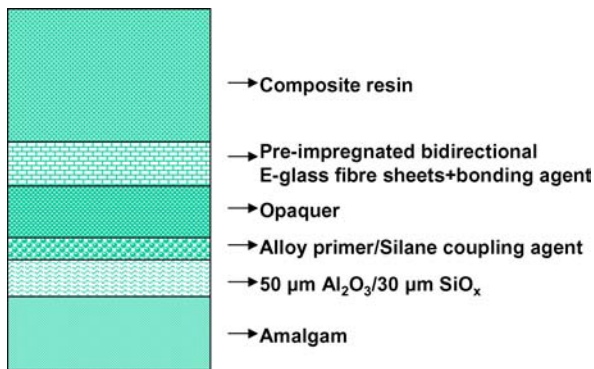


Figure 1 Schematic representation of the amalgam-composite specimens with corresponding surface conditioning methods.

ESPE AG, Seefeld, Germany) (Fig. 2). Following air-particle abrasion, a 3-methacryloxypropyltrimethoxy silane coupling agent (MPS) (ESPE[®]-Sil, 3 M ESPE AG) was applied and waited for its evaporation for 5 min.

In the glass fiber treated groups, two pieces of polymer-monomer gel pre-impregnated photopolymerizable bidirectional E-glass-fiber sheets (thickness: 0.06 mm, StickTech, Turku, Finland) were cut (diameter of the circular sheet: 3.6 mm) and placed in one group on the non-conditioned amalgam surfaces (Group 4) and in two groups on silica coated and silanized substrates (Group 5 and 6) (Fig. 3). Adhesive resin (Schotcbond Multipurpose Adhesive, 3M Dental Products, St Paul, MN 55144) was applied after the placement of each fiber sheet and it was light-polymerized (Optilux 501, Kerr, West Collins Orange, CA 92867) for 10 s. The polished amalgam surfaces were considered as control group (7).

In order to mimic the clinical situations where the amalgam is exposed and interfere with cosmetic-esthetic perspective, except group 5, opaquer (Visio-gem, 3M ESPE AG) was applied a thin layer in all groups and light-polymerized for 20 s.

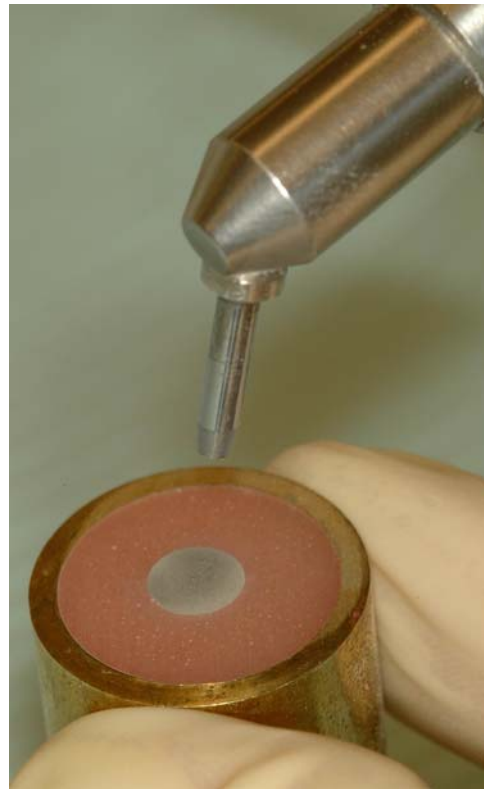


Figure 2 Application of chair side airborne particle abrasion on the amalgam surfaces using the intraoral sandblaster.

Bonding procedures

The bonding procedures were carried out in accordance with the manufacturers' instructions by the same operator throughout the experiments. The highly filled (79 wt% filler) resin composite (Tetric Ceram, Shade A2, Ivoclar, Schaan, Liechtenstein) was bonded to the conditioned amalgam specimens using translucent polyethylene molds with inner diameter of 3.6 mm and height of 5 mm. The resin composite was packed against the substrate with a composite-filling instrument. The resins were light polymerized for 40 s. Light-intensity was 770 mW/cm². Polyethylene molds were gently removed from the test specimens.

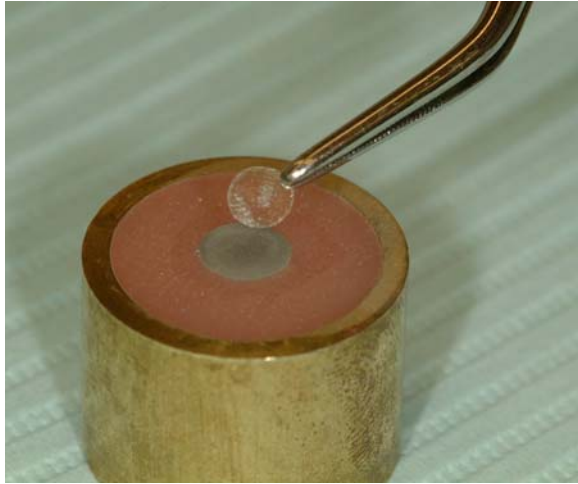


Figure 3 Application of polymer-monomer gel pre-impregnated photopolymerizable bidirectional E-glass-fiber sheets on the amalgam surfaces. The thickness of each fiber layer is 0.06 mm.

In an additional study ($n = 18, 6/\text{group}$) the mean surface roughness depth (R_Z) from the polished and air-abraded amalgam surfaces either with Al_2O_3 or SiO_x was measured (Perthometer S8P 4.51, Feinprüf GmbH, Göttingen, Germany). The mean roughness value was calculated from 3 single measurements. Each value represented the distance between the lowest and the highest point of the surface profile. These specimens were not used for the bond test in case the measurements may damage the surfaces.

All experimental groups were assessed at both dry and thermocycled storing conditions. While dry samples were kept in a desiccator at room temperature for 24 hrs prior to testing, the other groups were subjected to thermocycling (Thermocycler 2000, Heto-Holten A/S, Allerød, Denmark) for 6,000 cycles between 5°C and 55°C in deionised grade 3 water. The dwelling time at each temperature was 30 s. The transfer time from one bath to the other was 2 s.

Specimens were mounted in a jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, CA 94583) of the universal testing machine (Lloyd LRX, Lloyd Instruments Ltd., Lloyd, Canada) and a shear force was applied to the adhesive interface until fracture occurred. The specimens were loaded at a crosshead speed of 1.0 mm/min and the stress-strain curve was analysed with Nexygen 2.0 software.

Statistical analysis was performed using SAS System for Windows, release 8.02/2001 (Cary, NC). The comparisons between surface conditionings at dry conditions were made by one-way analysis of variance (ANOVA) with multiple comparisons using Tukey's honestly significant difference test. Student's t -test was used to determine the differences between dry and thermocycled conditions and Pearson's correlation coefficient was used to evaluate the association between surface roughness and bond strength. P values less

than 0.05 were considered to be statistically significant in all tests.

Results

Surface roughness values for the non-conditioned control group ($R_Z \sim 0.14 \mu\text{m}$) and for air-particle abraded surfaces with either Al_2O_3 ($R_Z \sim 0.19 \mu\text{m}$) or SiO_x ($R_Z \sim 0.16 \mu\text{m}$) did not show significant differences ($p = 0.23$) (Fig. 4). Furthermore the surface roughness was not significantly correlated with the bond strength values (Pearson's correlation coefficient $r = 0.42$, $p = 0.09$).

One-way ANOVA showed that shear bond strength was significantly affected by conditioning methods ($p < 0.001$). Fig. 5 displays box plots of the bond strength values associated with surface conditioning techniques at dry and thermocycled conditions. In dry conditions, silica coating and silanization followed by fibre sheet application exhibited significantly higher results with ($14.8 \pm 5.6 \text{ MPa}$) and without opaquer ($23.6 \pm 6.9 \text{ MPa}$) than those of the groups conditioned with alloy primer ($2.2 \pm 0.7 \text{ MPa}$, $p < 0.001$), air-particle abrasion + alloy primer ($4.4 \pm 2.0 \text{ MPa}$, $p < 0.001$), silica coating + silanization alone ($6.2 \pm 0.8 \text{ MPa}$, $p = 0.009$) or non-conditioned group (1.4 ± 0.6 , $p < 0.001$).

Silica coating and silanization followed by additional fibre sheets with opaquer application ($23.6 \pm 6.9 \text{ MPa}$) increased the bond strength significantly compared to those of other groups (for group 5, $p = 0.007$ and for other groups, $p < 0.001$).

Thermocycling decreased the bond strengths significantly for all of the conditioning methods tested (group 1, $p < 0.001$; group 2, $p = 0.013$; group 3, $p = 0.002$; group 4, $p = 0.026$; group 5, $p = 0.002$; group 6, $p < 0.001$; group 7, $p < 0.001$) and the least favourable results were obtained with the alloy primer treated groups with ($4.4 \pm 2.0 \text{ MPa}$, $1.0 \pm 1.9 \text{ MPa}$) or without air-particle abrasion ($2.2 \pm 0.7 \text{ MPa}$, 0 MPa) at both dry and thermocycled conditions, respectively.

Discussion

Replacement of amalgam restorations is associated with loss of tooth tissue by progressive cavity enlargement and repeated insults to the pulp. Since repair of amalgam restorations with amalgam is not reliable, experience indicates that an adhesive approach should be considered. Moreover, for the repaired restoration to withstand functional loads, strong and durable bond is needed.

Roughening the amalgam surface can increase the surface area and facilitate mechanical interlocking of the adhesive. The results of this study suggest that roughening the amalgam surface by air-particle abrasion provide some micro-roughness that was also in

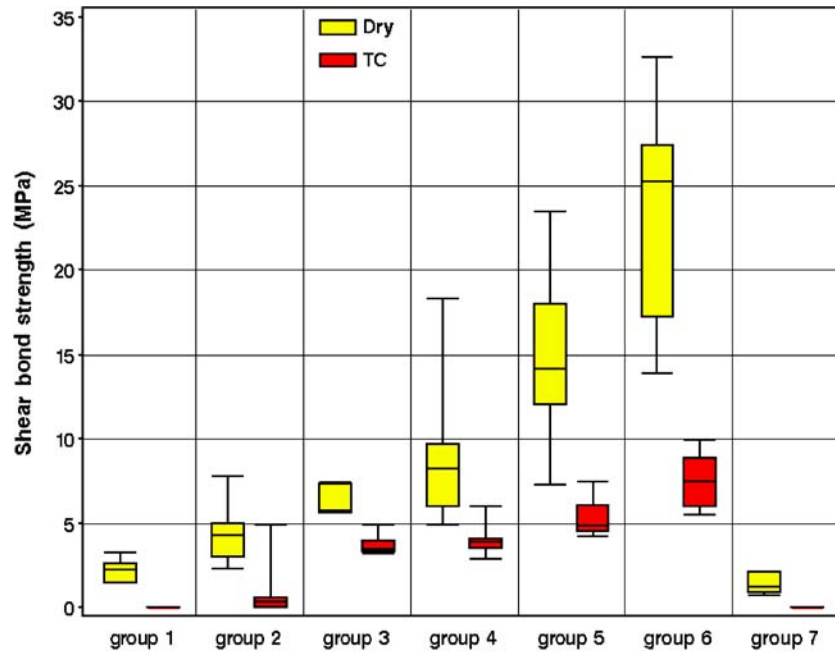


Figure 4 The surface roughness (μm) (R_z) of the tested amalgam ($n = 6$ per group) for air-particle abraded groups with either Al_2O_3 (Group 2) or SiO_x (Group 3) and non-conditioned group (Group 7). Box plots represent the minimum value, first quartile, median, third quartile and maximum value.

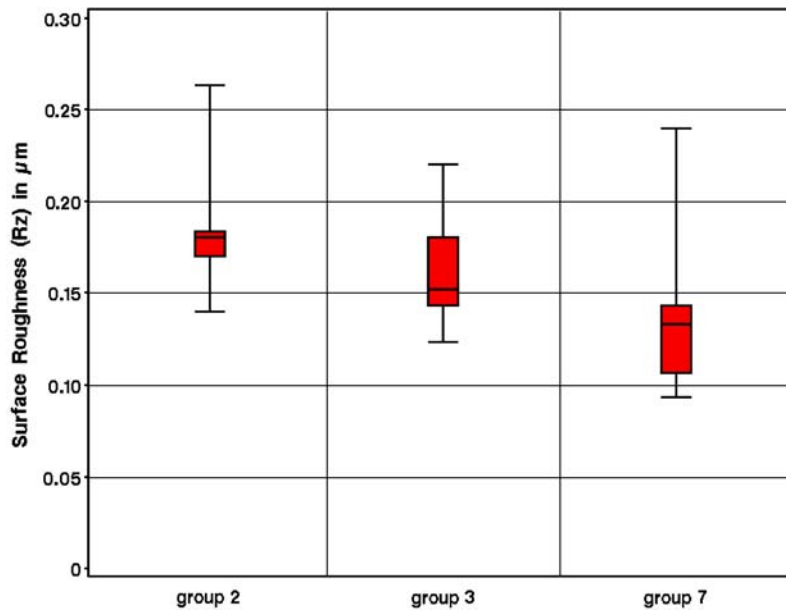


Figure 5 Shear bond strengths (MPa) of the resin composite bonded to conditioned amalgam substrates at dry and thermocycled conditions. Box plots represent the minimum value, first quartile, median, third quartile and maximum value.

accordance with several previous studies [2, 6, 12, 15, 16, 27] but in fact surface roughening itself did not dictate the bond strength. The present study was performed on fresh amalgam surfaces where a high surface energy can be expected but a change in surface roughness of high-copper amalgams over time, due to the formation of Cu_6Sn_5 crystals could also provide some roughness for micro-mechanical bonding.

Alloy primers are designed for conditioning both noble and base alloys and they are claimed to promote the bond strength. The postulated interaction mechanism of alloy primers involves adhesion to the alloy via hydrophilic carboxylate groups and the exposure of a hydrophobic moiety that is able to interact with the resin composite. The alloy primer used in this study contains VBATDT as the functional monomer for noble alloys

and hydrophobic 10-methacryloyloxydecyl dihydrogen phosphate monomer (MDP) as the functional monomer for base alloys [2, 8]. At present, the majority of the commercially available amalgam products are the high-copper amalgams. Use of these products avoids the formation of the easily corroding γ_2 (Sn₈Hg) phase. Amalgam is an alloy where mercury plays an essential role but the exact mechanism of adhesion to amalgam is unclear. Principally, silane (oligomers) monomers/molecules react with each other forming branched siloxane bonds, —Si—O—Si—, and with an inorganic substrate (matrix) (i.e. silica, metal oxides that contain basic hydroxyl —OH groups) with which they can form —Si—O—M— bonds (M = metal). It is likely that the oxide layer on the surface of the amalgam surface used in this study was not sufficiently formed as it is formed on other alloys. Therefore MPS silane coupling agent and MDP-VBATDT primer did not form durable covalent bonds with amalgams since there might not have been excess of hydroxyl groups on the surface. The type of amalgam may also influence the results. Higher bond strengths have been reported for bonding to spherical dental amalgam compared to lathe-cut or admixed amalgams [19]. However, in clinical practice it is not always possible for a clinician to determine the specific type of dental amalgam.

Tribochemical silica coating followed by silanization enhanced the bond between the resin composite and the amalgam compared with the non-conditioned or alloy primer treated groups. The quality and concentration of the surface oxides after silica coating and silanization affect the extent of molecular orientation providing a configuration that sterically favours cross-linking of the monomers of the resinous phase composites and thus increases the polymerization at the interface [29, 30]. However, no statistically significant differences were found in the bond strengths between the two air-particle abraded groups followed by either only alloy primer or MPS silane application.

One interesting finding of this study was the significant influence of the application of glass fibers on bond strength especially on the silica coated and silanized amalgam surfaces. The glass fibers used in this study were pre-impregnated with polymer-monomer gel. The results obtained in this group exhibited mean bond values ranging between 9.2 MPa and 23.6 MPa that exceeds the recommended ISO standard [31] and therefore could be considered strong enough for clinical applications. In general, stress concentrations within the resin and the interface can be relieved by initiation of a crack and its propagation through the resin until it meets the fibers, resulting in debonding of the resin composite. This phenomenon together with the interaction between the silica coated and silanized oxides needs further investigation on a large number of specimens. It is also possible that the polymer matrix between the glass fibers (semi-interpenetrating polymer network of

polymethylmethacrylate and cross-linked dimethacrylates) could have behaved as low modulus stress breaker between the amalgam and the repair composite resin.

The observation of a significant decline in bond strength after long-term thermocycling is probably due to the hydrolytic degradation of the chemical bond between the active monomers in the coupling agents studied and the amalgam substrates. Either water sorption or thermally-initiated reorientation of the coating might cause stress relaxation [29]. Direct comparison with previous studies is difficult to make since they differed in storage conditions but our findings after thermocycling with the use of alloy primer alone or air-particle abrasion with Al₂O₃ followed by alloy primer application were lower than those reports where specimens were tested either after short term water storage [15, 19, 32–34] or lower number of thermocycles [14]. After thermocycling, except for the glass fiber sheet treated groups, the bond strengths provided in other groups were lower than the recommended ISO standards.

In this study, opaquer has been advocated to mask the amalgam prior to composite bonding in order to simulate the clinical situations where fracture of amalgam was encountered with esthetics such as in some visible areas of the mouth. The opaquer used in this study was dimethacrylate based that is provided in a powder-liquid system. One can anticipate that bond strengths may vary with chemical composition and consistency of the opaquer. Lack of information also exists on the influence of the thickness of opaquer layer on the bond strength. It could be expected that the cohesive strength of the opaquer is lower than that of resin composite. Therefore a thick layer of opaquer might decrease the bond strength. However, this needs further investigation.

In clinical situations where an amalgam fracture is experienced, factors such as existence of intact enamel/dentin, repair resins with different elastic modulus, surface chemical composition, morphology and age of the amalgam could also affect the adhesion of resin composites to amalgam surfaces.

Conclusions

From this in-vitro study, the following conclusions were drawn:

1. Bond strengths of resin composite to amalgam substrates varied in accordance with the surface conditioning techniques.
2. Combination of silica coating and silanization with addition of optional pre-impregnated bidirectional e-glass fiber sheets at the adhesive interface increased the bond strengths significantly and therefore can be considered as an alternative method to improve attachment of resin composite to amalgam.

3. Thermocycling decreased the bond strength values substantially after all surface conditioning methods tested.

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