Bond Strength Comparison of Amalgam Repair Protocols Using Resin Composite in Situations With and Without Dentin Exposure

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Clinical Relevance

For reliable repair of amalgam restorations, including dentin fractures, the amalgam surface should first be silica coated; dentin/enamel should be etched, washed and rinsed thoroughly. Then, amalgam should be silanized and primer/bonding should be applied onto dentin.

SUMMARY

The replacement of defective amalgam restorations leads to loss of tooth material and weakens the tooth, creating an increased risk of cusp

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fracture. The repair of such defects is a minimal intervention technique. The current study compared the repair bond strengths of a resin composite to amalgam and an amalgam-dentin complex after various surface conditioning methods. The specimens (N=50) consisted of sound human canines with cylindrical preparations (diameter: 2.3 mm, depth: 3 mm) with amalgam-dentin complex (N=30, n=10/per group) and two groups with amalgam only (N=20, n=10/per group). The teeth were embedded in auto-polymerized polymethylmethacrylate (PMMA). The preparations were filled with non-Gamma 2 amalgam. The enamel was removed to expose dentin. The specimens with the amalgam-dentin complex were randomly assigned to one of the following conditioning methods: Group 1: Silicacoating amalgam, etching dentin, silane application on amalgam, primer/bonding on dentin, opaquer on amalgam, resin composite on both; Group 2: Etching dentin, silicacoating amalgam, silane

application on amalgam, primer/bonding on dentin, opaquer on amalgam, resin composite on and Group 3: **Etching** dentin. primer/bonding on dentin, opaquer, resin composite. The specimens with only amalgam were assigned to one of the following conditioning methods: Group 4: Silicacoating, silane application, opaquer, resin composite and Group 5: Opaquer, resin composite. For the two control groups, where no dentin was involved (Groups 4 and 5), bonding was achieved only on amalgam and Group 5 had no conditioning. The specimens were kept in water at 37°C for five weeks before bond strength (MPa ± SD) testing (Universal Testing Machine). After debonding, the failure types were analyzed. The results were significantly affected by the surface conditioning method (ANOVA). Only dentin conditioning (Group 3) showed the highest bond strength (39.9 ± 14) . The unconditioned control group (Group 5) showed the least favorable results (1.4 ± 0.5). Multiple comparisons (Tukey-Kramer adjustment) showed that the mean values of Group 1 (34.1 \pm 11.4), 3 (39.9 \pm 14) and 4 (35.5 \pm 4) were not significantly different (p>0.05), but between Groups 2 (22.8 \pm 6.6) and 3 (39.9 \pm 14), significant differences were observed (p=0.0027). For reliable repair of amalgam restorations, including dentin fractures, the amalgam surface should first be silica coated, then dentin/enamel should be etched, washed and rinsed thoroughly. Finally, the amalgam should be silanized and primer/bonding applied onto the dentin.

INTRODUCTION

In restorative dentistry, especially in the posterior region, amalgam is still commonly used. One of the problems associated with amalgam restorations is complete or partial fracture of cusps and/or the amalgam, itself, in the posterior region. With repair of the defective areas, it is possible to preserve both the dental tissues and restorative material, as removing parts of sound enamel or dentin while removing the whole restoration is almost inevitable. When removing dentin and enamel is avoided, strength is maintained and longevity of the tooth is increased. The risk of cusp fracture increases proportionally with the volume of the restoration. Minimal intervention techniques cause less destruction to tooth material compared to those of conventional techniques, with a reduced risk of tooth fracture and insults to the pulp during drilling.23

There is little information in the literature regarding the incidence of cusp and amalgam fractures. In a previous study on 46,394 patients, information on cusp fracture from 28 Dutch general practitioners over a three-month period was obtained.4 The incidence of cusp fractures was 20.5 per 1,000 persons in a year. For each new case of complete cusp fracture, the clinicians recorded information using a standard form, with questions related to location of the fracture, cause of fracture and restorative status of the tooth prior to the cusp fracture. Two-hundred and thirty-eight cases of complete cusp fractures were recorded. The molars were more frequently registered with cusp fractures than the premolars (79% vs 21%). The maxillary molars presented more fractures of the buccal cusps (66% vs 34%), while the mandibular molars presented more fractures of the lingual cusps (75% vs 25%). Nearly 77% of the cases had been restored on three or more surfaces. Mastication was most frequently reported as the cause of fracture (54%). In another study, the incidence rates for complete coronal fractures for all teeth were 89.0 and 72.7 per 1,000 personyear risk for all fractures and non-carious fractures, respectively. The rates for anterior and posterior teeth were 10.2 and 69.9, respectively. In mandibular posterior teeth, the lingual cusps were reported to fracture twice as frequently as the facial cusps, while the opposite was true for maxillary premolars. Among the maxillary molars, the mesiofacial and distolingual cusps fractured most frequently. A vast majority of fractures involved dentin exposure (95%), while pulpal exposure occurred less frequently (3%). A lower percentage of fractures extended below the gingival crest (24%) or the dentino-enamel junction (25%).⁵ In a long-term evaluation of extensive restorations in permanent teeth, the most frequent reasons for failure were fracture of the restoration (8%), secondary caries (6%) and fracture of the cusps (5%), regardless of the restoration type.6

There seems to be a number of reasons that contribute to tooth fracture, such as impact load, occlusal instability, fatigue load during mastication, secondary caries, technical errors, insufficient sound tooth material available surrounding the restoration or occlusal premature contacts. The more surfaces restored and the larger the dimensions of the preparation, the greater the risk of cusp fracture. The majority of fractures were supragingival, indicating that, in most cases, repair of the fractured teeth was not difficult. When restorations are repaired, there is minimal intervention to tooth structure, and the repair is more cost beneficial than replacement of the whole restoration. The supplementary of the supplementary of the structure.

Today, the most frequently used dental amalgam type is corrosion-resistant high-copper amalgams, which are available in one- and two-phase mixtures. Although amalgam is a reliable restoration material that may serve for many years, it cannot fulfill all cosmetic-esthetic demands. One of the reasons for replacing a failed amalgam restoration for an esthetic resin restoration, instead of repairing the amalgam restora-

tion, is poor esthetic appearance due to the reflection of amalgam under the resin composite. This problem is solved with veneering amalgam restorations with opaque resin materials. 12-13 Several repair techniques have been developed to repair fractured teeth with amalgam restorations. These repair techniques are based on either mechanical and/or chemical adhesion techniques. 7,13-17 In mechanical techniques, retention of the new restorative material is based on creating undercuts, grooves or the use of pins;18 whereas chemical adhesion techniques use adhesive agents to create a chemically stable interface to adhere the restorative materials. 19-20 Currently, alloy-resin bonding techniques also rely on both micro-mechanical and chemical adhesion principles. Numerous intra-oral repair systems are available, and a growing number of new systems are being introduced. Modern surface conditioning methods mostly require air-borne particle abrasion of the metal prior to bonding. These systems also involve conditioning the substrate, using bifunctional silane molecules that adhere to the metal surface after being hydrolyzed to silanol. Silanol groups form a polysiloxane network on the substrate, which finally reacts with the monomers of the opaquer and resin composite. 21 Unfortunately, there is no standard protocol described for the repair of amalgam restorations either in the literature or in the manufacturers' instructions, leaving clinicians doubtful in repair situations. It was reported that dentin tubules may be obstructed by the sand particles used in air abrasion devices, which lead to poor adhesion of resin composites.²² On the other hand, etching and rinsing is also

reported to diminish adhesion on alloy surfaces after air abrasion and silanization.

Therefore, the objectives of the current study were to assess different conditioning methods for cases where the amalgam-dentin complex and amalgam alone are present and to propose a repair protocol for defective amalgam restorations.

METHODS AND MATERIALS

The brand name, manufacturer, chemical composition and batch number of the materials used in the current study are listed in Table 1. The experimental groups, number of specimens and conditioning sequences are schematically presented in Figures 1a and 1b.

Specimen Preparation

The specimens (N=50) consisted of three groups (n=30)with amalgam-dentin complex and two groups (n=20) with amalgam only. Thirty recently extracted sound human maxillary canines without restorations or carious lesions were collected, cleaned of debris, had roots removed at the cemento-enamel junction and were embedded in auto-polymerized polymethylmethacrylate (PMMA) (Palapress, Vario, Hereaus Kulzer, Germany). PMMA was poured into a mold of a polyvinyl chloride tube (PVC, Martens, Netherlands) (diameter: 19 mm, height: 10 mm). The buccal side of each tooth was positioned upwards. In the center of the embedded teeth, an undercut cavity was prepared with a diameter of 2.3 mm and a depth of 3 mm using diamond burs (no: BRWH001S023, Komet, Brasseler, Lugano, Switzerland). In 20 other

Product Name	Company Name	Chemical Composition	Batch # F42028
РММА	Palapress, Vario, Hereaus Kulzer, Hanau, Germany	Polymethylmethacrylate	
Clearfil SE Bond Primer	Kuraray Co, Ltd, Tokyo, Japan	MDP, HEMA, Hydrophilic dimethacrylate, Camphorquinone, N,N- Diethanol p-toluidine, water	413277
learfil SE Bond Bonding	Kuraray Co, Ltd, Tokyo, Japan	MDP, Bis-GMA, HEMA, Hydrophobic dimethacrylate, Camphorquinone N,N- Diethanol p- toluidine, Silanated colloidal silica	413277
CoJet-Sand	3M ESPE AG, Seefeld, Germany	Aluminum trioxide particles coated with silica, particles size: 30 µm	165092
ESPE-Sil 3M ESPE AG, Seefeld, Germany		3-methacryloxypropyltrimethoxysilane, ethanol	152745
Visio-Bond 3M ESPE AG, Seefeld, Germany		Bisacrylate, aminodiol methacrylate, camphor-quinone, benzyl dimethyl ketale, stabilizers	260950

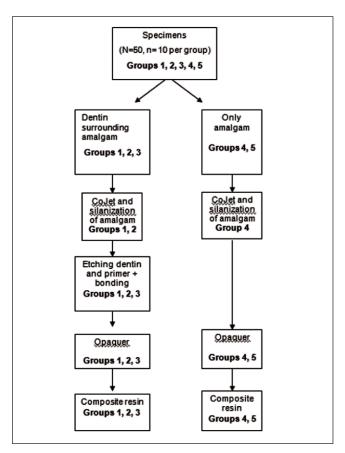


Figure 1a. Distribution of the experimental groups and sequence of surface conditioning methods employed.

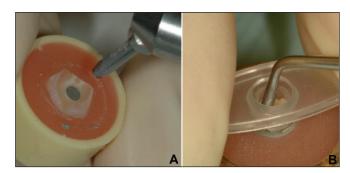


Figure 2. Figure 2a: Conditioning the amalgam by silica coating using an air-abrasion device, Figure 2b): packing resin composite incrementally on the substrate.

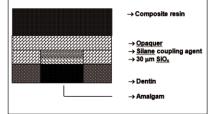


Figure 1b. Schematic representation of the applied methods on the amalgam-dentin complex in resin bonding.

PVC tubes, PMMA was poured and an undercut cavity (diameter: 6 mm, depth: 2-3 mm) was prepared with a diamond bur (no: H1S010, Komet, Brasseler).

A non- γ_3 amalgam (Cavex, Haarlem, The Netherlands) was triturated according to the

manufacturer's recommendations for six seconds in a high-energy mixer (Capmix ESPE, Seefeld, Germany) and condensed with hand instruments (Ash 49 and Ash 208-209, Dentsply, Surrey, United Kingdom) in the prepared cavities until they were slightly overfilled. The dentin surfaces were exposed using a trimmer (Rotogrind, Reitel Feinwerktechnik GMBH, Bad-Essen, Germany). The surface of each specimen was then ground finished to 1200 grit silicone carbide abrasive (Struers RotoPol 11, Struers A/S, Rodovre, Denmark). After grinding the specimens, they were cleaned for 10 minutes in an ultrasonic cleaner (Quantrex 90 WT, L&R Manufacturing, Inc, Kearny, NJ, USA), which was filled with deionized water.

Surface Conditioning Methods

The specimens in the three experimental groups with amalgam-dentin complex (N=30, n=10 per group) were randomly assigned to one of the following conditioning methods (Table 2): Group 1: Silica coating amalgam, etching dentin, silane application on amalgam, primer/bonding on dentin, opaquer on amalgam, resin composite on both, Group 2: Etching dentin, silica coating amalgam, silane application on amalgam, primer/bonding on dentin, opaquer on amalgam, resin composite on both and Group 3: Etching dentin, primer/bonding on dentin, opaquer, resin composite.

The specimens with only amalgam (N=20, n=10 per group) were assigned to one of the following conditioning methods: Group 4: Silica coating, silane application, opaquer, resin composite and Group 5: Opaquer, resin composite. In the two groups where no dentin was involved (Group 4, Group 5), bonding was

Table 2:	Table 2: Experimental Groups and Sequence of Surface Conditioning Methods								
Groups	N	Dentin Surrounding Amalgam	Silicatization of Amalgam (ESPE Sil)	Etching Dentin, Primer + Bonding (Clearfil SE Bond)	Opaquer (VisioGem)	Resin Composite (Clearfil Photobond)			
1	10	+	2	1	3	4			
2	10	+	-	1	2	3			
3	10	+	1	2	3	4			
4	10	-	1	-	2	3			
5	10	-	-	-	1	2			

achieved only on amalgam, while one group (Group 5) had no conditioning. These groups acted as the control groups.

Silica coating was performed using a chairside air abrasion device (CoJet, 3M ESPE AG, Seefeld, Germany) filled with CoJet-Sand (30 μ m Al₂O₃ coated with SiO₂, 3M ESPE AG) at a pressure of 2.5 bar from a distance of approximately 10 mm for five seconds (Figure 2a).

Only the dentin surfaces were etched with 35% phosphoric acid (Ultraetch, Ultradent Products, Inc, South Jordan, UT, USA) for 15 seconds and subsequently rinsed thoroughly with water. Silane coupling agent (ESPE-Sil, 3M ESPE AG) was applied only on the amalgam surface. After waiting three minutes for the silane reaction, both the primer and bonding agent were applied only on the dentin. Primer (Clearfil SE Bond Primer, Kuraray, Tokyo, Japan) was applied for 20 seconds and, subsequently, the bonding agent (Clearfil SE Bond Bonding, Kuraray) was applied for 10 seconds and photopolymerized for 20 seconds. The light intensity was 450 mW/cm² (SDS Kerr, Orange, CA, USA). After polymerization of the bonding agent on dentin, a thin layer of the opaquer (Visiogem, 3M ESPE AG) was applied on the amalgam surface and photopolymerized for 60 seconds.

Repair Composite Application

The resin composite (Clearfil Photo Posterior, Kuraray) was bonded to the amalgam and dentin surrounding the amalgam by use of translucent polyethylene molds (inner diameter: 3.6 mm and height: 5 mm). The bonding procedures were carried out in accordance with the manufacturers' instructions by the same operator (MÖ) throughout all of the experiments. The resin composite was packed against the substrate incrementally using a composite filling hand instrument (Ash-49, Dentsply) (Figure 2b) and photopolymerized in two layers of not more than 2 mm. Each layer was polymerized for 40 seconds from a distance of 2 mm. The light intensity was 450 mW/cm² (SDS Kerr). After polymerization, the polyethylene molds were gently removed from the test specimens. All the specimens were stored in water for five weeks at 37°C prior to testing.

Testing Procedure and Failure Analysis

After water storage, the specimens were mounted in the jig of a universal testing machine (Zwick Roell Z 2.5 MA 18-1-3/7, Ulm, Germany). Shear force was applied as close as possible to the surface of the substrate at a crosshead speed of 1 mm/minute until fracture occurred. The stress-strain curve was analyzed using Zwick Roell software (Zwick Roell, Ulm, Germany). Then, digital photos (Canon Ixus 40, Canon Inc, Tokyo, Japan) were taken from the substrate sur-

faces, and the types of failures were classified by interpreting the amalgam surface for cohesive or adhesive failures and the amount of opaquer that adhered to the amalgam.

A scoring system was established as follows: Score 0: no opaquer adhered to amalgam (0A) or cohesive failure in amalgam (0C); Score 1: less than one-third of the opaquer adhered to amalgam; Score 2: one-third to two-thirds of the opaquer adhered to the amalgam surface and Score 3: more than two-thirds of the opaquer adhered to the amalgam surface.

Statistical Analysis

Statistical analysis was performed using SPSS 11.0 software for Windows (SPSS Inc, Chicago, IL, USA). One-way Analysis of Variance (ANOVA) was used to analyze the means of each group. After ANOVA, the Tukey-Kramer's post-hoc test was performed, since the outcome of the different surface conditioning methods was statistically significant. Multiple comparisons were made to determine the effects of the different surface conditioning methods. *P*-values less than 0.05 were considered to be statistically significant in all tests.

RESULTS

Bond Strength

One-way ANOVA showed significant effects of conditioning methods on the bond strength results (p<0.001). Multiple comparisons (Tukey-Kramer adjustment) showed that the mean values of Groups 1 (34.1 ± 11.4), 3 (39.9 ± 14) and 4 (35.5 ± 4) were not significantly different (p>0.05); however, between Groups 2 (22.8 ± 6.6) and 3 (39.9 ± 14), significant differences were observed (p=0.0027) (Table 3, Figure 3).

The control group with no conditioning (Group 5) showed the least favorable results (1.4 ± 0.5 MPa), that of being significantly lower than the other groups (p<0.0001 for Groups 1, 3, 4 and p=0.0011 for Group 2). The group with only dentin conditioning (Group 3) showed the highest mean bond strength (39.9 \pm 14 MPa) but also the highest standard deviation.

Failure Analysis

Classification of the failure types per group is presented in Table 4. In Group 1, seven specimens showed adhesive failures between the opaquer and amalgam, with no opaquer left behind (0A) (Figure 4a), and three specimens showed mixed failures, where a small amount of opaquer still adhered to the amalgam surface (Score 1) (Figure 4b). In Group 2, four specimens showed adhesive failure between the opaquer and amalgam (Score 1), from which, in one specimen, nearly all the opaquer adhered to the amalgam surface (Figure 4c). While Groups 3 and 4 demonstrated only cohesive failures in the amalgam (0C), Group 5 had

Table 3: p-values of Multiple Comparisons, Tukey-Kramer Adjustment								
	Group 1	Group 2	Group 3	Group 4				
Group 1								
Group 2	0.0873							
Group 3	0.6587	0.0027						
Group 4	0.9981	0.0973	0.9048					
Group 5	<0.0001	0.0011	<0.0001	<0.0001				

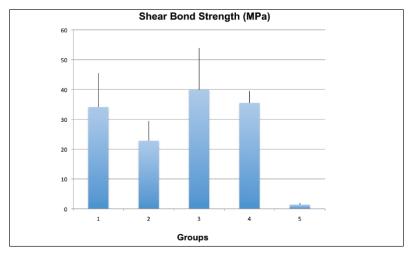


Figure 3. Mean repair bond strength and standard deviations of the five test groups after various surface conditioning methods.

exclusive adhesive failures between the amalgam and opaquer (0A), meaning that there was no opaquer left on the amalgam surfaces in all specimens.

DISCUSSION

Repairing a fractured amalgam restoration, instead of removing the entire restoration, is a minimally invasive approach in dentistry, since removal of a restoration is often associated with loss of tooth tissues as a consequence of cavity enlargement. A clinical problem is experienced in repair actions, where the use of multiple steps of adhesion promoters, which may contaminate one another in areas where two substrates, such

Classification of Failure Types According to the Scoring System Group 1 Group 2 Group 3 Group 4 Group 5 1 0A 0A 0C 0C 0A 0A 0C 2 0A 0C 0A 0A 0A 0C 3 0C 0A 0A 3 0C 0C 4 0A 5 0A 1 0C 0C 0A 0C 0C 0A 6 1 1 7 3 0A 0C 0C 0A 8 0A 0A 0C 0C 0A 0C 0C 9 1 0A 0A 10 0A 1 0C 0C 0A

 $0A=Adhesive\ between\ amalgam\ and\ composite,\ 0C=cohesive\ in\ amalgam,\ 1=\le 1/3,\ 2=\ge 1/3\ -\le 2/3,\ 3=\ge 2/3\ opaquer\ left\ on\ amalgam\ surface$

as amalgam and dentin, need to be conditioned. For this reason, in the current study, different sequences of repair protocols on the amalgamdentin complex were studied. The repair strength of resin composite to amalgam-dentin was compared with repair strength to amalgam only in order to find the best sequence. An opaquer has been advocated to mask the amalgam before applying the resin composite, since, in clinical situations, a fractured cusp could be in a visible area of the mouth. The major difference between paste opaquers and powder-liquid opaquers is their viscosity. 15,23-24 The opaquer used in the current study was a dimethacrylatebased powder-liquid opaquer. Considering the majority of failure types in all groups, it could be stated that the cohesive strength of opaquer was lower than the adhesion to amalgam.

The chairside tribochemical silica coating method, using CoJet-sand followed by silanization with an MPS silane, enhanced the bond strength between the resin composite and amalgam and dramatically enhanced the amalgam-dentin complex when compared with the nonconditioned group. However, there were no significant differences found between the other groups. The lower mean bond strength in Group 2 and the attributed to electrostic of the

2 could be attributed to obstruction of the dentin tubuli with silica particles, because, in this group, air abrasion was applied after etching. The obstruction of the dentin tubuli prevents penetration of the primer-bonding layer and formation of a hybrid layer, leading to lower bond strength. This supports the findings in Group 3, because higher bond strength values were obtained in this group. Since there was no air abrasion performed in this group, most probably, the dentin was not contaminated with sand particles.

There is limited information in the dental literature on the bond strength of repaired amalgam²⁵ and no study documents the bond strength of repaired amal-

gam involving dentin. The bond strengths in the current study were higher than the results presented in a previous study on amalgam-resin bond strengths (14.17 MPa). The specimens in the previous study, however, were aged differently from those in the current study.²⁵

Failure type classification is an essential part of bond analysis. For instance, in Group 5, no opaquer remained adhered to the amalgam surface after debonding, implying that the bond strength on amalgam in this group was lower than that in Groups 1 and 2. This is most probably due to no

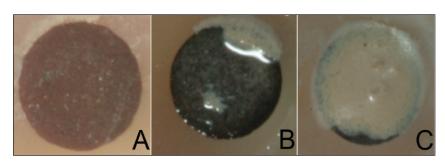


Figure 4. Representative photos of failure types after debonding, Figure 4a: adhesive failure at the interface (0A) (Score 0: no opaquer adhered on amalgam); Figure 4b: a combination of adhesive and cohesive failure in the opaquer (Score 1: ≤1/3 opaquer adhered) and Figure 4c: cohesive failure in the opaquer (Score 3: ≥2/3 opaquer left adhered on amalgam surface).

chemical adhesion existing between amalgam and the opaquer in the absence of silica-coating and silane application. On the other hand, in Group 3, higher standard deviations were obtained, which could be due to variations in dentin depth. In contrast to the expectations of the authors of the current study, no significant difference was observed in mean bond strength between silica-only coated and silanized amalgam (Group 4) and silicatized amalgam surrounded by dentin (Groups 1 and 2). The influence of dentin surrounding amalgam on bond strength could be further investigated in groups with different surface ratios of dentin and amalgam.

In the current study, in order to optimally simulate the clinical situation, dentin surrounding amalgam in a circular form was implemented, which could be considered an ideal model. In clinical situations, however, the shape and amount of dentin surrounding the fractured amalgam restorations could vary, thus affecting the longevity of the repair. Nevertheless, the results clearly showed the importance and necessity of conditioning amalgam and not relying merely on the bond strength that could be derived from tooth tissues. In the current study, the repaired amalgam was fresh. In clinical situations though, an amalgam restoration often requires repair after many years of service. In the oral environment, the corrosion of amalgam may yield to a microscopically rough surface.²⁶ This aspect requires further investigation.

Shear bond testing is a convenient method for screening the strength of adhesive interfaces *in vitro*. ¹⁹⁻²⁰ In the current study, where two substrates next to each other had to be tested, this test method could be considered ideal, compared to other test methods. However, one should consider that, in clinical situations, pure shear deformations are not likely to occur and adhesive strength of the bonded joints depends on a wide variety of parameters, such as the type of repair resin, type of amalgam, dentin morphology, aging and more. Among these factors, aging of the resin bonds can be simulated with long-term water storage and thermocycling. This is an important parameter, since

adhesive interfaces are weakened as a consequence of hydrolytic degradation of the coupling agent.^{23-24,27-28} In this study, the specimens were kept in water for only five weeks and no long-term water storage or thermocycling was performed. The results need to be verified after long-term aging conditions.

CONCLUSIONS

Based on this study, the following could be concluded:

- face). 1. Conditioning the amalgam surface dramatically increases the bond strength of resin composite for the repair of amalgam fractures with and without surrounding dentin.
- 2. The silica coating of amalgam after etching the dentin significantly diminished the bond strength results. Thus, the silica coating must be applied prior to etching the enamel/dentin.
- 3. An opaquer layer seems to be the weak point of the adhesion to amalgam.

Note

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