Abstract: The study aimed to compare the repair bond strength of aged composite and amalgam repaired with resin composite after various mechanical and adhesive surface treatments. Specimens were aged by thermal cycling (10,000 cycles, 5-55°C) and randomly subjected to one of three surface treatments: diamond bur abrasion, aluminum oxide air abrasion, or silica coating. Conventional bonding or a universal adhesive with incorporated silane was applied afterward (each \( n = 16 \)) and resin composite was attached. In the control groups (each \( n = 16 \)), resin composite was attached using one of the above adhesives without prior mechanical surface conditioning. After further thermal cycling, the shear bond strength (SBS) and failure modes were assessed. Statistical analyses were performed using ANOVA, Weibull statistics, two sample t-tests, and Chi²-test (\( P < 0.05 \)). The SBS of the repaired amalgam was significantly lower than that of the composite and mechanical pretreatment significantly increased SBS. The universal adhesive significantly improved the SBS of the repaired amalgam compared to the conventional bonding agent and mechanical pretreatment increased the number of cohesive/mixed failures. Amalgam restorations may be repaired using resin composites, but the resulting SBS is lower than that obtained with composite.

Keywords: amalgam; composite; dental restoration failure; dental restoration repair; shear bond strength.

Introduction

Although the repair of partially defective dental restorations has gained increasing attention over the past few decades, clinical studies on the success of restoration repair compared to newly placed or replaced restorations are limited. A recent review (1) revealed only five reliable studies on the repair of composite (2-5) and amalgam (2-4,6,7) restorations. The studies demonstrated that repaired amalgam restorations had a higher risk of failure than repaired composite restorations (1). Further findings suggested that repairs due to fracture of the original restoration had a worse prognosis than repairs due to secondary caries. This might be explained by the fact that amalgam restorations are more likely to fail due to fracture than composite fillings and might experience subsequent fractures when repaired (3). Another possible cause is a lower repair bond strength on amalgam compared to composite.

Repair techniques are often evaluated \textit{in vitro} based on bond strength measurements. The bond strength achieved after repair can be assessed with the shear bond strength (SBS), tensile bond strength, or mold-enclosed SBS (ME-SBS), each representing specific stress distributions.
(8) that make comparison difficult. While many studies have investigated the repair bond strength of composites (9-19), only a few have analyzed the SBS of repaired amalgam (20-23). A direct comparison of repaired composite and amalgam under the same conditions has yet to be performed. Comparison of the existing studies is also difficult due to high variability in study conditions, e.g., different aging conditions (13-15,19,23) and different mechanical (9,11-14,16,19,20,22) or adhesive surface treatments (9,10,12-14,16,18-20,23).

The following study was performed to compare the effectiveness of amalgam and composite restoration repair using various techniques to determine whether the lower survival of the former under clinical conditions can be partly attributed to lower repair bond strength.

As universal adhesives are known to be capable of bonding to various substrates (18), the possibility of improving the repair bond strength of composite and amalgam restorations using a universal adhesive containing silane and 10-methacryloyloxydecyldihydrogen phosphate (10-MDP) was assessed.

The null hypotheses were that there is (1) no difference in the repair bond strength of amalgam and composite repairs, (2) no effect from surface pretreatment on the repair bond strength of both substrates, and (3) no difference in the effect of different bonding agents on the repair bond strength of both substrates.

Materials and Methods

All procedures were performed by the same person. The study design is shown in Fig. 1.

Specimen preparation

Composite specimens (n = 128, diameter 6 mm, height: 3 mm) were prepared by placing resin composite (Filtek Supreme XTE, 3M ESPE, St. Paul, MN, USA; shade A2 body, lot: N779140) into brass hollow cylinders (inner diameter: 6 mm, height: 3 mm). Each increment was photo-polymerized (B.A. OPTIMA 10 LED, B.A. International, Hamburg, Germany) for 20 s at a distance of 2 mm from the surface. Light irradiance (>800 mW/cm²) was verified after the preparation of every 32 specimens using a radiometer (Cure Rite Model 644726, Dentsply Caulk, Milford, DE, USA). The composite specimens were water-stored for 24 h before further processing.

Amalgam specimens (n = 128) were prepared by condensing amalgam (Amalcap Plus Regular Nr. 3, Ivoclar Vivadent, Schaan, Lichtenstein; lot: VS0015) into the same brass hollow cylinders described above. The amalgam was triturated for 10 s using a capsule mixing device (CapMix, 3M ESPE). The amalgam specimens were water-stored for 24 h before further processing.

The specimens were not removed from the brass cylinders throughout the experiment. The enclosed specimens were flattened and polished using sandpaper of different grits (WS flex 18C, grits 500, 800, 1,200, Hermes, Hamburg, Germany; grit 4,000, Buehler, Esslingen, Germany) while being cooled with water (RotoPol-35 and PdM-Force-20, Struers, Willich, Germany). Diamond bur abrasion was simulated using sandpapers with 500-grit as adhesion studies in dentistry usually employ surfaces grinded with abrasive papers ranging between 60 and 1,200-grit (24). Polishing was accomplished using a custom-made mold that could hold four specimens at a time.

Only specimens without macroscopically visible irregularities were included in the subsequent experiments. Prior to repair, all specimens were artificially aged by thermal cycling (10,000 cycles, 5-55°C, dwell time: 20 s, transfer time: 10 s, Thermocycler V 2.9, Witytec, Gräfelfing, Germany).

Surface conditioning

For 24 h after thermal cycling, the specimens were stored in artificial saliva containing mucin, lysozym, α-amylase, and albumin in phosphate buffered saline (PBS) (25) to simulate protein coverage of the surfaces as present in the clinical situation. The surfaces were then dried using cotton pellets and wiped with alcohol for 5 s. For further processing, the specimens were randomly assigned to one of four groups subjected to the following mechanical surface conditioning techniques (each group n = 32):

- Surface roughening: To simulate abrasion with a
diamond bur, the surfaces were roughened using sandpaper (WS flex 18C, grit 500, Hermes) while being cooled with water. As the final step, the surfaces were thoroughly rinsed with water and then air blown.

- Air abrasion with aluminum oxide: The surfaces were sandblasted for 4 s with 50 µm aluminum oxide particles (Hager & Werken, Duisburg, Germany; lot not supplied by manufacturer) at an angle 45° to the surface and a distance of 10 mm with an air pressure of 2.2 bar. The remaining sandblasting agent particles were removed by air blowing.

- Silica coating: The surfaces were sandblasted for 4 s with 30 µm silica coated aluminum oxide (CoJet, 3M ESPE; lot: 630975) at an angle 45° to the surface and a distance of 10 mm with an air pressure of 2.2 bar. The remaining sandblasting agent particles were removed by air blowing.

- Control group: No further mechanical surface conditioning was applied.

Following surface pretreatment, all specimens were randomly conditioned with one of two different bonding agents: Scotchbond Universal (3M ESPE; lot: 630141) or Adper Scotchbond Multi-Purpose Plus Adhesive (3M ESPE; lot: N767654), each n = 16. The composition of the adhesives used is shown in Table 1. The adhesives were applied with a disposable microbrush for 20 s according to the manufacturer’s recommendations, gently air blown for 5 s, and light-cured for 10 s. A conventional bonding agent (Adper Scotchbond Multi-Purpose Plus Adhesive) was used without prior application of a primer, which is only necessary for the conditioning of dental hard tissue.

**Application of repair composite**

Repairs were performed with resin composite (Filtek Supreme XTE, 3M ESPE; shade A2 body, lot: N779140). Plastic hollow cylinders (inner diameter: 3 mm, height: 4 mm) were attached as molds on top of the substrate, filled with the repair composite in 2 mm thick increments, and light-cured for 40 s from a distance of 2 mm.

All specimens were subjected to an additional thermal cycling procedure after repair (10,000 cycles, 5-55°C, dwell time: 20 s, transfer time: 10 s).

**Measurement of mold-enclosed shear bond strength and failure analysis**

The repair bond strength was determined based on the mold-enclosed shear bond strength (ME-SBS) using a universal testing machine (Materialprüfmaschine 1446, Zwick, Ulm, Germany). Shear force was applied using a chisel-shaped loading device. The head moved at a speed of 1 mm/min parallel to the bonding surface. Using the software testXpert (12.1, Zwick), the ME-SBS σ was calculated from the maximum load with the debonding F (N) and adhesive area A (mm²): \( \sigma = \frac{F}{A} \).

Additionally, failure mode analysis was performed using a stereomicroscope (Stemi SV 11, Zeiss, Oberkochen, Germany) at 16× magnification as previously described (16-18). Failures that occurred only at the bonding interface were considered adhesive, while those that affected only the substrate or repair composite were considered cohesive. Failures were classified as mixed.

### Table 1 Composition of adhesives used

<table>
<thead>
<tr>
<th></th>
<th>Adper Scotchbond Multi-Purpose Plus Adhesive [Wt%]</th>
<th>Scotchbond Universal [Wt%]</th>
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</thead>
<tbody>
<tr>
<td><strong>Monomers</strong></td>
<td>- BISGMA (60-70)</td>
<td>- HEMA (15-25)</td>
</tr>
<tr>
<td></td>
<td>- HEMA (30-40)</td>
<td>- BISGMA (15-25)</td>
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<tr>
<td></td>
<td></td>
<td>- decamethylene dimethacrylate (5-15)</td>
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<tr>
<td></td>
<td></td>
<td>- DMAEMA (&lt;2)</td>
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<tr>
<td></td>
<td></td>
<td>- 10-MDP (unknown)</td>
</tr>
<tr>
<td><strong>Initiators</strong></td>
<td>- camphorquinone</td>
<td>- camphorquinone (&lt;2)</td>
</tr>
<tr>
<td></td>
<td>- amines</td>
<td>- dimethylaminobenzoa(-4) (&lt;2)</td>
</tr>
<tr>
<td><strong>Solvents</strong></td>
<td>none</td>
<td>- ethanol (10-15)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- water (10-15)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- butanone (&lt;0.5)</td>
</tr>
<tr>
<td><strong>Others</strong></td>
<td>- triphenylantimony (&lt;=0.5)</td>
<td>- silane treated silica (5-15)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- 2-propenoic acid, 2-methyl-, reaction products with 1,10-decanediol and phosphorous oxide (P205) (1-10)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- copolymer of acrylic and itaconic acid (Vitrebond Copolymer) (1-5)</td>
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<tr>
<td></td>
<td></td>
<td>- 2,6-di-tert-butyl-p-cresol (&lt;0.5)</td>
</tr>
<tr>
<td><strong>Viscosity</strong></td>
<td>0.25 Pa s</td>
<td>&lt;0.3 Pa s</td>
</tr>
</tbody>
</table>

Composition of adhesives used and viscosity as described in manufacturer’s Safety Data Sheets and resources. 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; BISGMA: Bisphenol A diglycidyl ether dimethacrylate; DMAEMA: (dimethylamino)ethyl methacrylate; HEMA: 2-hydroxyethyl methacrylate.
when both adhesive and cohesive failures occurred.

Statistical analysis
Statistical analyses were performed using the software IBM SPSS Statistics for Macintosh (version 24.0.0.1, Armonk, NY, USA). Each group was tested for normality using the Kolmogorov-Smirnov test and Lilliefors Significance Correction. Only two of the 18 groups were not normally distributed. Parametric tests were performed and the results were analyzed by three-way ANOVA using substrate (composite or amalgam), the kind of surface treatment, and the bonding agent applied as the factors.

Significant differences between various surface treatments were analyzed using one-way ANOVA and Scheffe’s (homogeneous variances) or Tamhane’s (non-homogeneous variances) post hoc tests. Two sample t-tests were used to determine significant differences between the applied bonding agents within the same surface treatments. Two sample t-tests were also used to analyze differences between the substrates.

Additionally, Weibull modulus $m$ and characteristic bond strength $\sigma_0$ (Weibull distribution parameters) were assessed using the maximum likelihood estimates and 95% confidence intervals (MathWorks MATLAB, version R2016b, Natick, MA, USA).

The effects of the substrate, kind of surface treatment, and bonding agent on the mode of failure were analyzed with the Chi$^2$ test.

The overall level of significance was set at $P < 0.05$.

Results
As it pertains to ME-SBS, the three-way ANOVA

<table>
<thead>
<tr>
<th>Table 2 Results of three-way ANOVA</th>
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<tbody>
<tr>
<td>Source</td>
</tr>
<tr>
<td>Corrected model</td>
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<tr>
<td>Intercept</td>
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<tr>
<td>Substrate</td>
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<td>Bonding agent</td>
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<td>Surface treatment</td>
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<td>Substrate * Bonding agent</td>
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<td>Substrate * Surface treatment</td>
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<td>Bonding agent * Surface treatment</td>
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<tr>
<td>Substrate * Bonding agent * Surface treatment</td>
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</table>

Results of three-way ANOVA evaluating the effect of the substrate (composite or amalgam), the applied bonding agent (Adper Scotchbond Multi-Purpose Plus Adhesive or Scotchbond Universal), and the kind of surface treatment.

![Fig. 2 Mold-enclosed shear bond strength (ME-SBS) in the composite and amalgam specimens treated with various repair techniques. Boxes represent median with interquartile range and whiskers represent the 5th-95th percentile. Outliers (<Q1-1.5*IQR or >Q3+1.5*IQR) are marked with an asterisk (*). Within groups conditioned with Adper Scotchbond Multi-Purpose Plus Adhesive, small letters indicate significant differences between mechanical surface treatments. Within groups conditioned with Scotchbond Universal, capital letters indicate significant differences between mechanical surface treatments. Significant differences between both adhesives within the same mechanical surface treatment groups are marked with †.](image-url)
revealed all factors and interactions except the interaction between the substrate and the bonding agent ($P = 0.225$) to be significant ($P \leq 0.001$). For details, see Table 2.

ME-SBS for all groups is presented in Fig. 2. Generically, composite repairs presented significantly higher ME-SBS than the respective amalgam groups ($P < 0.02$). All mechanical surface treatments showed a significant increase in ME-SBS compared to the respective control group. There was no significant difference between the bonding agents in the composite group, with the exception of the control group and group treated with roughening. All amalgam specimens treated with Scotchbond Universal showed significantly higher ME-SBS than the corresponding groups conditioned with Adper Scotchbond Multi-Purpose Plus Adhesive. Nonetheless, even silica coating and application of Scotchbond Universal led to lower repair bond strength for the amalgam compared to the composite specimens.

The Weibull distribution parameters and their 95% confidence intervals are shown in Table 3. For both substrates, the lowest characteristic strength was observed in groups not subjected to mechanical pretreatment or those treated with mechanical roughening when the conventional bonding agent was used. Composite specimens subjected to mechanical treatment and the application of Scotchbond Universal showed the highest characteristic strength ranging from 27.2 to 29.0 MPa. Amalgam specimens presented lower characteristic strength than the respective composite groups (up to 23.8 MPa). The Weibull modulus $m$ ranged from 0.2 to 9.4 for amalgam specimens.

The distribution of failure types differed significantly depending on the substrate, surface treatment, and bonding agent ($P < 0.001$). For composite specimens, the failure types changed from adhesive to mixed and...
particles, which might further increase the surface area and allow for the formation of Si-O-Si-bonds when silane-containing adhesives are used (30).

Chemical surface conditioning was performed with a conventional bonding agent or a universal adhesive with incorporated silane. Differences were observed between both adhesives for all amalgam groups and for composite specimens, which were not treated mechanically or by roughening. This observation can be partly explained by the higher viscosity of Adper Scotchbond Multi-Purpose Plus Adhesive compared to Scotchbond Universal, which can be attributed to the higher amount of BISGMA. The lower viscosity of Scotchbond Universal might result in better wettability of the substrate and thus, higher bond strength compared to specimens treated with Adper Scotchbond Multi-Purpose Plus Adhesive. The silane incorporated into Scotchbond Universal might have further enhanced ME-SBS on aged composite specimens without optimal mechanical pretreatment (i.e., only roughened or even untreated surfaces) by forming bonds to exposed filler particles. ME-SBS on composite specimens pretreated by air abrasion or silica coating could not be further enhanced by the application of Scotchbond Universal. Adhesion to these surfaces may have been already optimized by mechanical pretreatment with no further potential for improvement by the application of a universal adhesive. On amalgam surfaces, silane allows for the formation of Si-O-bonds on aged and therefore oxidized amalgam surfaces. Even after abrasion of the superficial layer by mechanical pretreatment, oxidized amalgam is present in the subsurface layer (31). Additionally, Scotchbond Universal contains 10-MDP, which is an adhesive monomer capable of bonding to non-noble metals such as amalgam (35,36).

Composite specimens showed significantly higher ME-SBS in all groups and even optimal surface conditioning of amalgam did not result in comparable ME-SBS. Higher ME-SBS of composite specimens compared to metal restorations was confirmed in a recent study and allow for the formation of Si-O-Si-bonds when silane-containing adhesives are used (30).

Discussion
Although the use of amalgam for the placement of new dental restorations is increasingly rare (26,27), many amalgam restorations are still in service and, thus, there is a high demand for suitable amalgam repair procedures. This study showed that amalgam restorations can be repaired using composite. However, the repair bond strength is lower than that obtained with composite restorations and more dependent on the mechanical as well as chemical pretreatment applied.

In the present study, repair was performed after thermal cycling to simulate the time between original restoration placement and failure. After placement of the repair composite, additional thermal cycling was performed to assess the long-term stability. As opposed to water storage (aging due to water uptake only), thermocycling simulates hydrothermal aging with temperature changes resulting in repetitive contraction-expansion stresses (24,28). The number of cycles used (10,000 cycles) might correspond to approximately one year of clinical service (29). Thermal cycling affects composite and amalgam surfaces in different ways. For example, it removes free radicals that can react with the repair composite (10,30). The surfaces of zinc-free amalgams, which were used in the present study, are oxidized and present oxide layers primarily consisting of tin oxide (31). Therefore, the formation of covalent bonds between oxygen/OH-groups and Si atoms of silane-containing adhesives might be possible (23).

The repair techniques employed in this study (roughening, air abrasion, silica coating, and application of a bonding agent) are frequently utilized in dental practice (32,33). As found in previous studies, silica coating and air abrasion led to higher repair bond strength compared to mechanical roughening for both composite (9,12,16,30,34) and amalgam (20). Air abrasion and silica coating are assumed to result in a more regular and, thus, more retentive surface topography than the use of diamond burs. Silica coating might further improve repair bond strength due to the incorporation of silica particles, which might further increase the surface area and allow for the formation of Si-O-Si-bonds when silane-containing adhesives are used (30).

The materials used in the present study were brittle. Due to the variable existence of strength-controlling flaws, the overall repair performance can be better expressed with two parameters, the Weibull modulus $m$ and Weibull characteristic bond strength $\sigma_c$. A lower Weibull modulus $m$ reflects higher variability in the achieved bond strength and subsequently, lower reliability of the characteristic bond strength $\sigma_c$. Repair techniques with lower Weibull modulus $m$ are therefore likely to be more technique sensitive (37).

Even with optimal mechanical and adhesive surface
conditioning, the ME-SBS of amalgam was significantly lower than that of composite. Silica coating or air abrasion followed by the application of the universal adhesive resulted in bond strength values of approximately 20 MPa, which are assumed to be clinically sufficient (12). Therefore, while partially defective amalgam restorations can be repaired using resin composites, silica coating, and the application of a silane-containing adhesive are required to achieve optimal bond strength.

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Conflict of interest
None.

References


