Bond Strength between Amalgam and Tooth Hard Tissues with Application of Fluoride, Glass Ionomer Cement and Adhesive Resin Cement in Various Combinations

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The bovine enamel and dentin samples ground flat with a series of emery papers were pretreated with fluoride either before or after etching, and several of the dentin samples were lined with glass ionomer cement. Adhesive resin cement, Panavia or Super-Bond, was applied on the enamel and dentin samples, and then amalgam was placed on these samples.

The highest bond strength between amalgam and enamel was 90 kg/cm² for Panavia, and 102 kg/cm² for Super-Bond. These values were obtained by the pretreatment with Ag(NH₃)₂F. The bond strength between amalgam and the dentin treated with fluoride or the etching agent was 13-17 kg/cm² for Panavia, and 23-65 kg/cm² for Super-Bond. The bond strength between amalgam and lined dentin was 41-46 kg/cm² when Panavia was used. The bond strength tended to be increased by the fluoride treatment prior to etching.

We suppose that the adhesion technique for amalgam would be clinically valuable.

Key words: Amalgam, Bonding, Adhesive resin cement

INTRODUCTION

In recent years adhesive composite resins are being used even for occlusal restorations of posterior teeth, but it is unlikely that the composite resins will totally replace silver amalgam within the foreseeable future. Amalgam has been used successfully for over a century and is more durable material than the composite resins when it is used to fill occlusal cavities of posterior teeth. However, since amalgam is not adhesive to tooth structures, microleakage normally occurs around the freshly placed restoration.

If amalgam remains non adhesive, it may in time be replaced by the adhesive composite resin in almost all cases. If, however, we develop suitable adhesion technique for amalgam restorations, amalgam would become more useful and more important posterior restorative material than in the past.

This study was done to determine the bond strength between amalgam and the tooth tissues using fluoride, glass ionomer cement and adhesive resin cement.

MATERIALS AND METHODS

Freshly extracted bovine incisors, stored in saline after extraction, were used. A tooth block having the enamel or dentin surface of approximately 8×8 mm was cut from the labial part of each tooth. The enamel and dentin surfaces were ground flat using a grinding and polishing machine with a series of emery papers and finished with 600 grade. Then each
tooth block was trimmed so as to fit into the round window (10 mmΦ) of a metal mold. The block was embedded in the metal mold with acrylic resin except for the flattened experimental surface.

Table 1 shows the etching agents, fluorides, glass ionomer cements and resin cements used. Table 2 and 3 show the treatment procedures for enamel samples and dentin samples, respectively. The fluorides used were 8% SnF₂, 38% Ag(NH₃)₂F (Saforide) and acidulated 2% NaF (Fluor N). The fluoride was topically applied with a cotton pellet for 3 min to the experimental surfaces. The lining was placed on several dentin samples with glass ionomer cement in a thickness of 200 μm.

A copper band (inside diameter 6.5 mm) was attached to the experimental surface with

Table 1  Etching agents, fluorides, cements for lining, and resin cements used in this study

<table>
<thead>
<tr>
<th>Material</th>
<th>Code</th>
<th>Batch</th>
<th>Manufacturer</th>
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</thead>
<tbody>
<tr>
<td>Etching Agents</td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Clearfil etching agent*</td>
<td>E</td>
<td>ET 355</td>
<td>Kuraray</td>
</tr>
<tr>
<td>Super-Bond etching agent for dentin**</td>
<td>10-3</td>
<td>41201</td>
<td>Sunmedical</td>
</tr>
<tr>
<td>Fluorides</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stannous fluoride (8% SnF₂ solution)</td>
<td>SnF₂</td>
<td>V5H 4664</td>
<td>Nakarai Chemicals</td>
</tr>
<tr>
<td>Saforide (38% Ag(NH₃)₂F solution)</td>
<td>Ag(NH₃)₂F</td>
<td>401 AA</td>
<td>Toyo Seiyaku Kasei</td>
</tr>
<tr>
<td>Fluor N (acidulated 2%NaF solution)</td>
<td>NaF</td>
<td>409 AA</td>
<td>Toyo Seiyaku Kasei</td>
</tr>
<tr>
<td>Cements for lining</td>
<td></td>
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<td></td>
</tr>
<tr>
<td>Lining Cement</td>
<td>Lining C</td>
<td>140561/230561</td>
<td>G-C</td>
</tr>
<tr>
<td>Ketac-BOND</td>
<td>Ketac B</td>
<td>00101/0009</td>
<td>ESPE</td>
</tr>
<tr>
<td>Resin Cements</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Panavia EX</td>
<td>Panavia</td>
<td>PN-0300/PL-208</td>
<td>Kuraray</td>
</tr>
<tr>
<td>Super-Bond C&amp;B</td>
<td>Super-Bond</td>
<td>50201/41001/433</td>
<td>Sunmedical</td>
</tr>
</tbody>
</table>

* 40% Phosphoric acid gel
** 16% Citric acid-3% ferric chloride solution

Table 2  The treatment procedures for enamel surfaces

<table>
<thead>
<tr>
<th>No.</th>
<th>Enamel Surface Treatment</th>
<th>Number of measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>E* (60sec) + Panavia</td>
<td>4</td>
</tr>
<tr>
<td>2</td>
<td>SnF₂ + E* (60sec) + Panavia</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>SnF₂ + Panavia</td>
<td>5</td>
</tr>
<tr>
<td>4</td>
<td>Ag(NH₃)₂F + E* (60sec) + Panavia</td>
<td>5</td>
</tr>
<tr>
<td>5</td>
<td>Ag(NH₃)₂F + Panavia</td>
<td>5</td>
</tr>
</tbody>
</table>

Super-Bond

<table>
<thead>
<tr>
<th>No.</th>
<th>Enamel Surface Treatment</th>
<th>Number of measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>10-3** (30sec) + Super-Bond</td>
<td>5</td>
</tr>
<tr>
<td>7</td>
<td>SnF₂ + 10-3** (30sec) + Super-Bond</td>
<td>5</td>
</tr>
<tr>
<td>8</td>
<td>NaF + 10-3** (30sec) + Super-Bond</td>
<td>5</td>
</tr>
<tr>
<td>9</td>
<td>Ag(NH₃)₂F + 10-3** (30sec) + Super-Bond</td>
<td>5</td>
</tr>
</tbody>
</table>

* Clearfil etching agent
** Super-Bond etching agent for dentin

† Ecomet III, Buehler, Evanston, USA
Table 3 The treatment procedures for dentin surfaces

<table>
<thead>
<tr>
<th>No.</th>
<th>Dentin Surface Treatment</th>
<th>Number of Measurements</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>E* (5 s) + Panavia</td>
<td>6</td>
</tr>
<tr>
<td>2</td>
<td>SnF₂ + E* (5 s) + Panavia</td>
<td>5</td>
</tr>
<tr>
<td>3</td>
<td>E* (5 s) + SnF₂ + Panavia</td>
<td>6</td>
</tr>
<tr>
<td>4</td>
<td>Ag(NH₃)₂F + Panavia</td>
<td>6</td>
</tr>
<tr>
<td>5</td>
<td>Lining C + E* (60 s) + Panavia</td>
<td>6</td>
</tr>
<tr>
<td>6</td>
<td>Ag(NH₃)₂F + Lining C + E* (60 s) + Panavia</td>
<td>6</td>
</tr>
<tr>
<td>7</td>
<td>Lining C + Ag(NH₃)₂F + E* (60 s) + Panavia</td>
<td>6</td>
</tr>
<tr>
<td>8</td>
<td>Ketac B + E* (60 s) + Panavia</td>
<td>6</td>
</tr>
</tbody>
</table>

* Clearfil etching agent
** Super-Bond etching agent for dentin

The bond strength between amalgam and the enamel was 82 kg/cm², and the bond strength was increased by the pretreatment with Ag(NH₃)₂F prior to acid etching (90 kg/cm²). However, when Panavia was applied to the enamel treated with SnF₂ or Ag(NH₃)₂F without etching, the bond strength showed a marked decrease (36% decrease in SnF₂, 66% decrease in Ag(NH₃)₂F).

For the measurement of bond strength, the specimen was mounted in an Instron Universal Testing Machine. All specimens were sheared at the tooth-amalgam interface, parallel to the enamel or dentin surface. A crosshead speed of 0.5 mm/min was used. The shear force at which amalgam was dislodged was measured with a strip chart recorder and calculated in kg/cm².

RESULTS

Figure 2 shows a graphic representation of the average shear bond strengths between amalgam and enamel. When Panavia was applied to the etched enamel, the average bond strength between amalgam and the enamel was 82 kg/cm², and the bond strength was increased by the pretreatment with Ag(NH₃)₂F prior to acid etching (90 kg/cm²). However, when Panavia was applied to the enamel treated with SnF₂ or Ag(NH₃)₂F without etching, the bond strength showed a marked decrease (36% decrease in SnF₂, 66% decrease in Ag(NH₃)₂F).

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When Super-Bond was applied to the enamel treated with the 10% citric acid-3% ferric chloride (10⁻³) solution, the average bond strength was 96 kg/cm², and the bond strength to the enamel was increased by the pretreatment with Ag(NH₃)₂F (102 kg/cm²). However, it was decreased when the enamel was pretreated with SnF₂ or NaF (46% decrease in SnF₂, 30% decrease in NaF).

Figure 3 shows the average shear bond strength between amalgam and dentin. When Panavia was applied to the dentin treated with the etching agent or fluoride, the average bond strength was 13-17 kg/cm². However, when dentin was lined with glass ionomer cement, the bond strength between amalgam and the dentin was 41-46 kg/cm², the highest bond strength (46 kg/cm²) being obtained by the use of Ag(NH₃)₂F treatment after the glass ionomer cement lining.

When Super-Bond was applied to the dentin etched with the 10⁻³ solution, the average bond strength was 56 kg/cm², and it was increased slightly by the fluoride treatment before etching with the 10⁻³ solution (61-65 kg/cm²). However, the bond strength was decreased greatly when SnF₂ treatment was given between 10⁻³ etching and Super-Bond application (23 kg/cm²).
Even adhesive restorative materials, not to mention nonadhesive traditional ones, always have a microscopic space between the restoration and the cavity walls. This microscopic space has been demonstrated by the use of radioisotope tracers, dyes, the microscope and other techniques.

Since under in vivo conditions, oral fluid, acids and microorganisms may penetrate freely into this microscopic space, the cavity walls would be in danger of secondary caries at all times. For the prevention against secondary caries, it would be very important not only to minimize microleakage itself but also to provide the cavity walls with an anticariogenic property. The mediation of resin cement and glass ionomer cement at the amalgam-tooth interface would reduce the microleakage, and the fluoride treatment of tooth tissues may increase the resistance to carious attack. In fact, the use of topical fluoride solution to the prepared cavity is known to enhance the carious resistance of tooth tissues of cavity walls and to arrest secondary caries.

In the meantime, the effects of the topical fluoride in bonding procedures have been studied by a number of investigators. The addition of sodium fluoride to the etching agent reduced greatly the resin-enamel bonding. The acidulated fluoride treatment, either before or after etching of enamel, markedly reduced the bond strength of the sealant to enamel. However, Low et al. examined the influence of acidulated phosphate fluoride and stannous fluoride on the adhesion of resin materials, and revealed that the bond strength increased slightly in only the combination of stannous fluoride and Nuva Seal. Kimura et
al.19) investigated the relationship between the addition of fluoride to the etching agent and the bond strength to enamel. They showed that the bond strength of Super-Bond was not affected by the different concentrations of the fluoride. Thus, a fluoride treatment does not always interfere with the bonding of the resin material to enamel.

The bond strength between amalgam and the enamel was increased rather when the enamel was treated with SnF₂ or Ag(NH₃)₂F before the application of Panavia, but it was decreased markedly when Panavia was applied without acid etching. When Super-Bond was used as an adhesive, the bond strength between amalgam and enamel was increased only by the pretreatment of Ag(NH₃)₂F and was decreased markedly by the other fluoride treatments. These results suggest that fluoride treatment is not always a contra-indication for enamel-resin cement bonding, and that the treatment should be done before the etching procedure. Moreover the proper combination of fluoride and resin cement should be used. The increased bond strength by fluoride treatment may be due, in part, to the inhibition of over-decalcification by the etching agent.

When Panavia was used as an adhesive between amalgam and dentin, the bond strength was considerably lower than that of Super-Bond. The reason for this would be that as the dentin surface has many openings of tubules which usually contain air in non vital teeth, the aerobic condition at the dentin-Panavia interface may interfere with sufficient curing of Panavia which is an anaerobic curing resin. When Super-Bond was used as an adhesive, the bond strength between amalgam and dentin increased by the pretreatment of SnF₂ followed by etching with the 10⁻³ solution.

Application of etching agents and resin materials directly to the dentin of vital teeth is not commonly recommended. For the protection of pulp tissues a lining is made with base materials such as calcium hydroxide or glass ionomer cement. Yokogawa et al.20) examined the bond strength between three types of base cements and two resin cements. The combination of glass ionomer cement and Panavia showed higher bond strength than the combinations of the other base cements and Panavia. We demonstrated that when Panavia was used between amalgam and the dentin lined with glass ionomer cement, the bond strength increased markedly in comparison with the direct application of Panavia to dentin. When Panavia was used as an adhesive, the highest bond strength between amalgam and dentin was obtained with the combination of Lining Cement + Ag(NH₃)₂F + Etching + Panavia.

Although many procedures and techniques to reduce microleakage of amalgam restorations have been reported21−24), there was no technique for complete sealing. Fukuda24) showed that when an anaerobic curing sealant was applied on the margin of amalgam restorations, the sealant penetrated well into the microspace between the amalgam and the cavity walls, and reduced the dye penetration at the margin of amalgam. Moreover he revealed that the artificial caries around the amalgam restorations followed by the application of the sealant was inhibited effectively. Judging from his results, the adhesion techniques for amalgam presented in our study may probably reduce the microleakage in the same way and inhibit secondary caries around the amalgam restorations.

Since Super-Bond begins to cure itself quickly after it is applied to the cavity walls, it may be difficult to use in a clinical situation. In contrast, Panavia never hardens before amalgam filling, because it is an anaerobic curing resin. Panavia, therefore, would be more
favorable than Super-Bond, if we intend to apply these techniques practically.

CONCLUSIONS

The shear bond strength between amalgam and the tooth hard tissues was examined after the application of fluoride, glass ionomer cement and adhesive resin cement in various combinations.

1) Amalgam was adhered to the tooth hard tissues using resin cement as an adhesive.
2) The bond strength tended to increase with the fluoride treatment prior to etching.
3) The highest bond strength between amalgam and enamel was 90 kg/cm² for Panavia, and 102 kg/cm² for Super-Bond.
4) The bond strength between amalgam and the dentin treated with fluoride or etching agent was 13-17 kg/cm² for Panavia, and 23-65 kg/cm² for Super-Bond.
5) The bond strength between amalgam and the dentin lined with glass ionomer cement was 41-46 kg/cm² when Panavia was used as an adhesive.
6) These results suggest that these adhesion techniques for amalgam restorations are very useful in the clinic.

REFERENCES


接着力レジンと合金の接着機構の解明に関する研究

——高温酸化層表面に存在する数分子層の吸着水による接着性低減化の実験的証明——

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東日本学園大学歯学部歯科理工学教室

前報において、Co-Cr 合金の高温酸化表面には吸着した数分子の水が存在しており、そのため酸化表面と 4-
META 側鎖との間に数分子層の水が介在していることが推定された。これが酸化表面に対する接着性が、研磨
したままで形成される不働態被膜に対するよりも劣っている原因と結論された。本報では、酸化層表面の吸着水
を脱水し、この表面に対する接着性を調べ、この結論の正しさを実験的に検証した。

先ず、Co-Cr 合金と 18-8 ステンレス鋼を 500℃、大気
中で酸化処理を行い、次に、1 × 10−4 Torr の減圧下で
700℃ 加熱し、酸化層表面の脱水を行った。接着操作は
水分の存在しない高純度アルゴンガス中で行った。液体
チッ素を用いた熱サイクルを加えた後、引張試験を行っ
た結果、2 つの合金とも酸化したままで全面酸化
とレジン内破壊が混在しているが、脱水処理後ではほと
んどがレジン内破壊を呈した。酸化層表面に吸着水が存
在しなければ優れた接着性を示すことが明らかになった。

歯質との接着に関する研究

その 2 アミド基を有するモノマーの合成とその歯質接着性

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*日本大学松戸歯学部歯科理工学教室 **日本大学付属歯科医工学専門学校

アミド基を官能基として有するモノマー、4-メタクリ
ロキシベンゾアミド (MBA)、4-メタクリロキシ
フェニルプロピオンアミド (MPPA) を合成し、その接
着性を調べた。対照として MMA、O-メタクリロイル
チロシンアミド (MTYA) を用いた。

被着体としては、新鮮牛歯生体歯、および象牙質を
用いた。エナメル質、象牙質ともに、10％クエン酸－3％
FeCl₃ 水溶液で 30 秒間処理した。各モノマーを MMA
に溶解し、TBB-O を重合開始剤として用いた。その結
果、次のような接着強さ (MPa) が得られた。MMA：
11.1±5.3（エナメル質）、5.7±2.8（象牙質）：MMA/
MPPA：12.1±6.0（エナメル質）、12.3±4.0（象牙質）：
MPPA/MMA：11.8±2.5（エナメル質）、12.1±3.5（象
牙質）：MTYA/MMA：11.8±3.5（エナメル質）、
14.1±6.1（象牙質）。

接着性レジンセメントの介在によるアマルガムと歯質との接着

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接着性レジンセメントをアマルガム-歯質間で介在させることにより、いかなる強さでアマルガムを歯質に接
着させることができるかを検討した。牛歯を用い、被
験面は酸による処理、フッ化物 (SnF₂，Ag(NH₃)₂F，
NaF) による処理を種々に組み合わせて前処理した。また
象牙質面ではグラスアイオノマーセメントでライニング
した場合についても調べた。

その結果、アマルガムとエナメル質の接着強さは、Ag
(NH₃)₂F で前処理した時最も高い値（パナビアで 90 kg/cm²,
スーパーポンドで 102 kg/cm²）が得られた。象牙
質に対してはスーパーポンドが高い接着強さ（23〜65
kg/cm²）を示した。パナビアの象牙質に対する接着強さ
コンポジットレジン修復の象牙質窩鍵からの微小漏洩に及ぼす
Cleaning Agents の効果

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われわれはこれまでにキレート剤、低濃度の無機・有機酸、あるいは象牙質有機質に対する効果を期待して、
取れん作用を持つ塩類などのなかから、生体に対してマイルドで象牙質清掃効果の高い処理剤（Cleaning
Agent）として使用できるものを模索してきた。今回は、それらのなかから、比較的低濃度で、被処理象牙質面の
SEM観察によっても良好な smear層の除去効果が認められた 10％アスコルビン酸、10％クロ酸、10％リン酸および3％乳酸アルミニウムを処理剤として用いて、
全象牙質窩洞への処理効果を辺縁封鎖性の評価によって
検討した。

各種の清掃剤の中では 10％クロ酸が最も高い処理
効果を示したが、窩洞側窩鍵からの微小漏洩を完全には
阻止することができないことがわかった。さらに、被処
理歯の条件をよく揃えた今回の実験でも、窩洞に辺縁
封鎖性に大きい差を生じることなどから、象牙質窩鍵の
封鎖の困難性が示唆された。

純チタンの既製ポストへの応用
——金属鍛造コア用ポストとしての適応性——

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チタンを既製ポストとして応用することを試み、本報
ではまず金属鍛造コア用ポストとしての適応性について
検討するために、機械的性質ならびにコア鍛造による影
響について現在市販されている Ni-Cr合金および18-8
ステンレス鋼の既製ポストと比較した。

純 Ti の素材線材は冷間引抜後焼なまし処理を施され
ており、機械的性質はほぼ金合金 Type IIIに相当する。
Ni-Cr合金と18-8ステンレス鋼ポストのかたさおよび
引張強さは Tiポストよりかなり大きいが、コア鍛造過
程での加熱によって焼きなましを受けて、かたさは Tiポ
ストを下回るようになり、強さにも大きな差は認められ
なくなった。しかし、純 Ti は 1,300℃まで加熱すると、
酸化などによって著しく劣化してしまう。Tiポスト
上への他合金コアの鍛造性は非常に良好であり、1,000℃
まではもちろんその影響もなかったことから、鍛込みが
1,000℃前後まで完了する合金コアに対しては、Tiポ
ストは有用であると考えられる。

充てん用コンポジットレジンに関する研究
第1報 光重合型コンポジットレジンの硬化特性

有川裕之*，藤井孝一*，蟹江隆人**，田畑泰治*，奥 淳一*，上新和彦*，井上勝一郎*，
内山長司***，黒木茂代子****