Surface Preparation of the Dental Au-Ag-Pd Alloy for Improving Adhesion to 4-META/MMA-TBB Resin Cement

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Comparisons of simple surface preparation of the dental Au-Ag-Pd alloy for improving the adhesion to 4-META/MMA-TBB resin cement were carried out by bonding metal to metal and metal to the etched enamel.

The anodized surface with phosphoric acid after sandblasting kept sufficient bond strengths during immersion in water at 37°C for three months, whereas that after cleansing showed a marked decrease in bond strengths during immersing times from one up to three months. Bond strengths of the surface only sandblasted were low in the early stage of water immersion but increased during immersing times from one up to three months.

It appeared that 4-META/MMA-TBB resin cement continuously formed new adhesion even during immersion in water.

Key words: Dental Au-Ag-Pd alloy, 4-META/MMA-TBB resin, Adhesion

INTRODUCTION

The introduction of resin cement adhering both to metal and to the tooth\(^1\)\(^2\) permits conservative teeth preparations for cast restorations, causing a marked change in the clinical technique\(^3\)\(^4\). For obtaining good adhesion, it is essential that the adhesive operation must be carried out immediately after the proper surface treatment according to resin and adherend used without contaminating the treated surface. A few methods of the surface treatment of the dental Au-Ag-Pd alloy for improving the adhesion to 4-META/MMA-TBB resin cement have been reported\(^5\)\(^6\)\(^7\). But the techniques unfortunately require masking and re-polishing when these surface treatments are performed after marginal rubbing, bite control and polishing, so that easier and readier clinical surface preparation is desired.

This study was carried out to investigate simple surface preparation of the dental Au-Ag-Pd alloy for improving the adhesion to 4-META/MMA-TBB resin cement.

MATERIALS AND METHODS

1. Measurement of bond strengths and arithmetical average roughness

   Experiment 1: Bonding between metals

   Plates (1.5×5.5×11.5 mm) and attachments (4 mm in diameter) for the tensile bond test were prepared by the usual centrifugal casting, using a dental 12% Au-Ag-Pd alloy of
Castwell M. C.*, and then cleaned twice ultrasonically in distilled water for 10 min. Half of them were immersed in a boiling cleanser** for Au-Ag-Pd alloy for 2 min to remove oxide formed after casting. After being washed with a stream of distilled water for 15s, the cleansed surface was dried with an air gun for 15s. To remove oxide formed after casting, the other half were sandblasted at 5 kgf/cm² from a distance of about 5 mm for 30s using Paasche's air brush. After being ultrasonically cleaned in distilled water for 10 min, the sandblasted surface was washed with a stream of distilled water for 15s and dried with an air gun for 15s. After adhesive tape was applied to the plates except a width of 6 mm in the central part, and around the attachments, to restrict the area for surface treatment, surface treatments as shown in Table 1 were carried out both on the plate and on the attachment using a brush for applying nitric acid and potassium permanganate, and using a brush of Ez-oxisor§§ for anodizing with ammonium peroxodisulfate and phosphoric acid.

After the surface treatments finished, plates and attachments were washed with a stream of distilled water for 15s and dried with an air gun for 15s, then the arithmetical average roughness of plates was measured using a Kosaka Surfcorder Model SE-3C#. The sampling length and cut off used were 4 mm and 0.8 mm, respectively. The plates and attachments were then bonded together by the brush on technique using Super Bond C & B##. The resin was allowed to set at room temperature for 10 min under a pressure of 0.8 kgf/cm². During this time, the excess soft resin was removed. After the resin set, adhesive tape was removed and the hardened excess resin was removed again. Then, after the specimens were immersed in water at 37°C for one week, tensile bond strengths were measured using a Digital Compressive and Tensile Testing Machine Type PS-200† at a cross head speed of 2.5 mm/min. The force needed to break a bond was recorded and expressed in kgf/cm². In addition to this tensile bond test, bond strengths of the specimen anodized with phosphoric acid were measured after further immersion in water at 37°C for one and three months. The same tests were repeated four times for each test both in the experiment 1 and 2. A schematic diagram of the experimental procedure is shown in Fig. 1.

Table 1  Surface treatments of metal and the enamel

<table>
<thead>
<tr>
<th>Metal</th>
<th>Enamel</th>
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<tr>
<td>(1) None</td>
<td>(1) 65% Phosphoric acid for 60 s</td>
</tr>
<tr>
<td>(2) 61% HNO₃ for 5 s</td>
<td>(2) 30% Citraconic acid for 60 s</td>
</tr>
<tr>
<td>(3) 1% KMnO₄ in 3% H₂SO₄ for 15 s</td>
<td></td>
</tr>
<tr>
<td>(4) Anodizing (DC 3V, ca. 3mA) with 10% (NH₄)₂S₂O₇ for 30 s</td>
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<tr>
<td>(5) Anodizing (DC 4V, ca. 20mA) with 40% H₃PO₄ for 30 s</td>
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* G-C Co., Tokyo, Japan
** Sankin Industry Co., Osaka, Japan
$ Paasche Airbrush Co., Chicago, USA
§§ Towagiken Co., Osaka, Japan
† Kosaka Lab. Co., Tokyo, Japan
## Sun Medical Co., Kyoto, Japan
† Tokyo Shikenki Seisakusho, Co., Tokyo, Japan
Experiment 2: Bonding of metal to the etched enamel

Sound human maxillary and mandibular premolars extracted for orthodontic treatment and stored in a cooled physiological saline solution were used. The root and lingual cusp side of the tooth crown were cut off and the facial cusp side was fixed with cyanoacrylate on a brass block that was used as an operating table for preparing the tooth, and the facial enamel surface was flattened with 400 grit silicon carbide paper under running water on a Wingo Ripe polishing machine*, until 500-900 \( \mu \)m from the most projected facial enamel was removed. Subsequently, the enamel surface was etched with 65% phosphoric acid, which was an appendix of Super Bond C & B, and with 30% citraconic acid8) for 1min, using a sponge, an appendix of Super Bond C & B. After being washed with distilled water for 15s using a spray of the three way syringe, the etched surface was dried with an air gun for 15s. Then, the etched enamel was bonded to the sandblasted attachments with or without phosphoric acid anodization in the same manner as in the experiment 1, and tensile bond strengths were measured after immersing in water at 37°C for one week, and for one and three months.

2. Analysis of metal elements with electron probe micro analyzer (EPMA)

Counts of metal elements on the cleansed and the sandblasted surface prepared in the

* Wingo Co., Osaka, Japan
same manner as in the experiment 1 were measured with an EPMA-810Q*. The metal surface in which large surface irregularity was formed using a diamond disk (No. 62) and the surface polished up with diamond paste (0.25 µm in diameter), were also analyzed to evaluate the influence of surface roughness. The measurement was repeated four times at different places in one specimen.

The accelerating voltage, specimen current and measuring time used were 20 kV, 5 nA and 10s, respectively.

3. Observation with scanning electron microscope (SEM)

The cleansed and the sandblasted surfaces prepared in the same manner as in the experiment 1 were observed after the surface treatment at the same area as had been observed before the treatment. The etched enamel prepared in the same manner as in the experiment 2, the debonded metal surfaces and the enamel surfaces after the tensile bond test were coated with Au-Pd in an Eiko Engineering Ion Coater Model IB-2** and also examined with a Hitachi S-430 SEM*** at 20 kV.

RESULTS

1. Measurement of bond strengths and arithmetical average roughness

Figure 2 shows results of the measurement for arithmetical average roughness. No

![Graph showing results of measurement for arithmetical average roughness.](image)

![Graph showing results of measurement for tensile bond strength.](image)

* Shimadzu Co., Kyoto, Japan
** Eiko Engineering Co., Ibaragi, Japan
*** Hitachi Co., Tokyo, Japan
The changes of tensile bond strengths of the specimen anodized with phosphoric acid after sandblasting and cleansing in metal to metal adhesion during immersion in water at 37°C for three months. Statistically significant differences were observed, although the surface, only sandblasted, tended to show a slightly higher value.

Figure 3 shows results of the measurement for tensile bond strength in metal to metal adhesion after immersing in water at 37°C for one week. The bond strengths obtained by every surface treatment used in this study were higher to the sandblasted surface than to the cleansed surface. The surface, only sandblasted, showed lower bond strength than the sandblasted surface oxidized with potassium permanganate and that anodized with phosphoric acid, but higher bond strength than that oxidized with nitric acid and that anodized with ammonium peroxodisulfate. The cleansed surfaces, received with any surface treatment, showed higher bond strength than the surface only cleansed. There were no statistically significant correlations between arithmetical average roughness and bond strengths.

Figure 4 shows the changes in bond strengths between metals, anodized with phosphoric acid, during immersion in water at 37°C for three months. The sandblasted surface, anodized with phosphoric acid, kept the bond strength of more than 200 kgf/cm² even after three months. Contrary to this, the bond strength of the cleansed surface, anodized with phosphoric acid, decreased extremely during immersing times from one up to three months.

Figure 5 shows the changes in bond strengths between metal and the etched enamel during immersion in water at 37°C for three months. Bond strengths of the surface, only sandblasted, were low at first but increased during immersing times from one up to three months, whereas those of the sandblasted surface, anodized with phosphoric acid, were constantly high with a tendency to decrease after three months.

Figure 6 shows the effect of enamel etching solutions on bond strengths. Citraconic acid
Fig. 6 The changes of tensile bond strengths for the enamel etched with 65% phosphoric and 30% citraconic acid in metal to the etched enamel adhesion during immersion in water at 37°C for three months.

Figure 7 shows the debonded metal surfaces that were after the tensile bond test between metal and the etched enamel. Fragments of the enamel attached on three of the surfaces that were only sandblasted and most of the surfaces without enamel fragments showed the interface failure at the metal surface (Fig. 7a), and on all sandblasted surfaces that were anodized with phosphoric acid (Fig. 7b), after immersing in water at 37°C for one week. Fragments of the enamel decreased with an immersion in water at 37°C for more than one month (Figs. 7a, b). But there increased the surfaces in which ring-shaped resin fragments attaching along the outside of the metal surface were observed, and the width of the ring tended to increase with an increase of the immersing period (Fig. 7a).
2. Analysis of metal elements with EPMA

Figure 8 shows results of the measurement for count of metal elements. No great differences were observed in counts between the roughened and the polished surface.

The count number of copper was very low, but that of gold and silver was high in the cleansed surface as compared with other surfaces. The sandblasted surface showed the higher count number of copper than the cleansed surface, but showed the lower count number of other elements than the other surfaces.

3. Observation with SEM

SEM photographs of the cleansed and the sandblasted surfaces before and after surface treatment are shown in Figs. 9-12. The cleansed surface showed wrinkle-like irregularities with many holes. The great majority of the holes ranged in diameter from about 0.5 µm to smaller. The sandblasted surface showed irregularities like a scratch of various sizes running to all directions, which were larger than irregularities of the cleansed surface.

The nitric acid treatment caused such a remarkable dissolution on the cleansed surface that aspects observed before the treatment became unrecognizable, and small holes became larger and partly connected with each other to form linear grooves. The sharp micro-feature of the sandblasted surface observed before the treatment became roundish, scratches being enlarged (Fig. 9).

After the treatment with potassium permanganate, some of the smallest holes observed before the treatment in the cleansed surface became unobservable with flat contrast of the overall view, and very small particles were observed on the sandblasted surface and the micro-feature of the surface underlying the particles became indistinct (Fig. 10).

After being anodized with ammonium peroxodisulfate, the cleansed surface showed the same observation as that obtained by the treatment of potassium permanganate, and the
Fig. 9  

a: SEM of the cleansed surface. (×4,000)  
b: SEM of the surface of Fig. 9a oxidized with HNO₃. (×4,000)  
c: SEM of the sandblasted surface. (×4,000)  
d: SEM of the surface of Fig. 9c oxidized with HNO₃. (×4,000)

sandblasted surface showed an indistinct micro-feature of the surface in the central part with flat contrast of the overall view (Fig. 11).

After being anodized with phosphoric acid, the surfaces showed the same observation of the overall view as that resulting from the anodization with ammonium peroxodisulfate, but a vague oxide film was observed clearly in the cleansed surface. Particles having existed before the treatment disappeared and new particles appeared at the different places after the treatment for the sandblasted surface (Fig. 12).

Clearer grooves at the prism periphery and more distinct crystals of the appatite were observed in the enamel etched with citraconic acid (Figs. 13a, b) than in the enamel etched with phosphoric acid (Figs. 13c, d).

Figure 14 shows SEM photographs of the third metal surface from the left of 1W and the fifth from the left of 3M as shown in Fig. 7a, and of their corresponding enamel surfaces.
Resin remained intact on the enamel surface but little attached to the metal surface after immersing in water at 37°C for one week (Figs. 14a, b).

Resin fragments, due to the cohesive failure of resin, attaching along the outside of the metal surface with little attaching at the central part were noted after immersing in water at 37°C for three months (Fig. 14c). A cohesive failure of resin was observed along the outside of the remaining resin on the enamel surface, tending to infiltrate into the central part (Fig. 14d).

DISCUSSION

The bond strengths obtained by each surface treatment used in this study were higher to the sandblasted surface than to the cleansed surface. Mechanically treated metal surfaces
possess exo-electron emission in relation to the defect and the transformation of metal crystals, which is related to the surface reactivity. Therefore, it appears that these phenomena affect the high bond strength of the sandblasted surface that the oxides after casting are mechanically removed.

Though no statistically significant differences were observed in the measurement of arithmetical average roughness, the sandblasted surface showed large surface irregularities as compared with the cleansed surface by the examination with SEM. Consequently, since the sandblasted surface has wider adhesive areas allowing resin to penetrate without entrapping air bubbles than those of the cleansed surface when the surface is treated with low reactivity to the adhesive monomer (4-META or 4-MET), surface irregularities are thought to affect the bond strength to some extent. However, no remarkable differences in aspects of the sandblasted surface were observed before and after being anodized with ammonium...
peroxodisulfate or phosphoric acid, but the bond strengths were very different from each other, so it appears that surface irregularities have little effects on the bond strength.

On the other hand, measurement with EPMA for counts of aluminium at the sandblasted surface showed about 17,000 counts under the same condition as in this study, suggesting the existence of plenty of alumina. This fact is thought to affect fewer counts of the sandblasted surface than those of the polished and the roughened surface in any metal element, although a state of the surface such as irregularities and porosity of the surface, and segregation of the metal element at the surface may influence the number of the counts. But effects of alumina remaining in the sandblasted surface on the bond strength are not clear. Moreover, the sandblasted surface contained more easily oxidizable copper with less hardly oxidizable silver and gold than the cleansed surface. The fact that the cleansed surface contained little copper agrees with the result of the previous study\(^9\) on the pickling of oxides formed on gold
alloys during high temperature oxidation, although the selective dissolution of copper in the surface during cleansing oxides may take part in this fact. The differences in contents of copper between the cleansed and the sandblasted surface appear to produce a marked effect on the bond strength for none treated surface that the oxides after casting were only removed and on adhesive durability during immersion in water.

The fact that the bond strength varied with each oxidizing treatment appears to depend on such differences in the oxide film as the reactivity to the adhesive monomer, thickness, mechanical strength and durability to water.

Such effects as electrolytic polishing, etching, cleaning and oxidation usually occur when metal is anodically polarized. Though the thickness and the mechanical strength of the oxide film vary with kinds of metal and solution used and the chemical nature of the oxide film formed, the oxide film in which incorporates anions existing in the solution is generally
formed on the metal surface in any case. The anodic oxidation with phosphoric acid is carried out in the aircraft industry for the pre-treatment of aluminium, so that a porous, less conductive and firm oxide film is being utilized for the adhesion to epoxy resin\(^1\). Chemical conversion treatments with phosphoric salts for increasing the adhesion between steel surface and coating are also carried out. Moreover, in adhesive dentistry, many so-called adhesive resins have phosphoric ester in their molecular structures. Consequently, the good adhesion obtained by anodizing with phosphoric acid appears to be due to the phosphide introduced into the oxide film.
Since the anodizing condition using 40% phosphoric acid at 4V DC for 30s had shown the best adhesion for the cleansed surface when acid concentrations, voltages and periods of the treatment were altered among phosphoric, citric and sulfuric acid, these conditions were adopted in this study. However, as great differences in adhesive property after immersing in water at 37°C for a long period existed in this study between the cleansed and the sandblasted surface, and considerably good adhesion had been obtained with citric acid, it appears to be necessary to re-examine the anodizing conditions for the sandblasted surface.

Such factors as adhesive force at the interface between adhesive and adherend, their cohesive force, the direction and the speed of the load and a shape of the test piece, are considered to affect the bond strength. As the enamel was frequently broken down, it seems that the bond strength of metal to the enamel was lower than that of metal to metal when the sandblasted surface was anodized with phosphoric acid.

After immersing in water at 37°C for one week, the enamel fragments attaching to the debonded metal surface were observed in the three surfaces only sandblasted, but in all the sandblasted surfaces anodized with phosphoric acid. It appears that the cohesive force of resin is stronger than that of the enamel at this time. Also, a few interface failures between metal and resin were observed in the surfaces only sandblasted by the examination with the naked eye, where few resin fragments were observed by the examination with SEM. It seems that the decrease in the number of enamel fragments after immersing in water at 37°C for more than one month is due to the increased cohesive failure of resin itself owing to the decreased cohesive force of resin caused by the absorption of water. Contrary to these findings, bond strengths of the surface, only sandblasted, increased during immersing times from one to three months.

Ring-shaped resin fragments attaching along the outside of the debonded surfaces, only sandblasted, became distinct after immersing in water at 37°C for more than one month, and the width of the ring increased with an increase of the immersing period. Moreover, there were few resin fragments in the central part of the debonded surfaces only sandblasted, and resin firmly attached to the corresponding enamel surfaces but showed the ring-shaped cohesive failure along the outside of the remaining resin on the enamel. And the cohesive failure of resin infiltrated into the central part by the examination with SEM. Therefore, the bond between metal and the enamel appears to be constructed through the ring, which is quite different from the case after immersing in water at 37°C for one week.

These findings suggest that copper copiously existing in the sandblasted surface is corroded, and a very small amount of the unpolymerized adhesive or resin monomer dissolving in water newly adheres to corroded copper. However, in addition to this hypothesis, alumina remaining in the sandblasted surface, the surface irregularity, and the ratio of 4-META and 4-MET in the monomer12) may also influence the new adhesion, so further investigations appear to be needed.

This new adhesion appears to be capable of maintaining the bond strength of more than 200 kgf/cm² even after immersing in water at 37°C for three months in metal to metal adhesion for the sandblasted surfaces that were anodized with phosphoric acid, and of increasing in the bond strength in metal to the enamel adhesion for the surfaces only sandblasted even during immersing times from one up to three months.
Remarkable differences in aspects were observed between the enamel surface etched with 30% citraconic and 65% phosphoric acid. Wider and deeper grooves at the prism periphery, which are necessary for resin to form a tag, and clearer small surface irregularities caused by the enamel apatites were observed in the enamel etched with 30% citraconic acid than in the enamel etched with 65% phosphoric acid.

In spite of these findings, although the bond strength after etching with 30% citraconic acid tended to be higher than that of 65% phosphoric acid, no statistically significant differences were observed between these acids in the bond strength. This fact suggests that the adhesion between the etched enamel and resin is strongly affected by other factors than an anchoring effect of the resin tag.

It is reported that 4-META/MMA-TBB resin possesses the adhesive property to the unetched enamel to some extent,1) the 4-MET to which 4-META hydrolyzes, contains the same adhesive properties as 4-META13) and an abundance of the adhesive monomer which is mostly converted into 4-MET is highly maldistributed in the resin reinforced dentin14). Furthermore, it is considered that the etching solution remains in the etched enamel in extremely small quantities15) and forms compounds with Ca16). Consequently, the high and durable bond strength of the enamel etched with 65% phosphoric acid appears to be due to the high adsorbability of the polar adhesive monomer to phospho-Ca compounds or crystals of the enamel apatite and the continuous formation of new adhesion to the enamel surface under relatively high acid condition12) as in the case of metal.

It is suggested that the adhesive mechanism between the resin cement used in this study and metal depends on the intermolecular or hydrogen bond5,17) between the adhesive monomer and metal oxides, so that lots of surface preparation with alloys for improving the adhesion are presented18-22). Though oxidation by heating5) and potassium permanganate in acid solution6), and plating with tin7) are reported for a dental Au-Ag-Pd alloy, these treatments require the troublesome technique such as re-polishing, drying with heat and masking. On the other hand, anodizing with phosphoric acid after sandblasting used in this study is thought to be simple and effective surface preparation because a clean surface can be obtained if this procedure is performed just before setting a dental casting, due to the cleaning and degreasing effect of anodizing, even if the sandblasted surface is contaminated by saliva and fingers at bite control or polishing, and the high and durable bond strength can be obtained after immersing in water at 37°C for a long period. Also, bond strengths of the surface, only sandblasted, tended to increase during immersion in water for a long period with low in the early stage, so that only sandblasting appears to be a considerably effective surface treatment if low bond strengths in the early stage can be reinforced in some way, and if the contamination of the sandblasted surface with saliva or fingers does not affect the adhesion23) of 4-META/MMA-TBB resin cement.

CONCLUSION

Comparisons of simple surface preparation of the dental Au-Ag-Pd alloy for improving the adhesion to 4-META/MMA-TBB resin cement were carried out by bonding metal to metal and metal to the etched enamel. Moreover, measurement of arithmetical average
roughness and counts of metal elements with EPMA, and observations with SEM were carried out. The following results were obtained.

1) The bond strengths obtained by any surface treatment used in this study were higher to the sandblasted surface than to the cleansed surface. The surface anodized with phosphoric acid both after cleansing and sandblasting showed the highest bond strength after immersing in water at 37°C for one week. The anodized surface with phosphoric acid after sandblasting kept sufficient bond strength during immersion in water at 37°C for three months, whereas that after cleansing showed a marked decrease in bond strengths during immersing times from one up to three months. Bond strengths of the surface, only sandblasted, were low in the early stage of water immersion but increased during immersing times from one up to three months.

2) No statistically significant differences were observed in any surface preparation for arithmetical average roughness and no correlations between the tensile bond strength and arithmetical average roughness.

3) Measurement with EPMA revealed that the sandblasted surface appeared to contain much copper but little silver and gold as compared with the cleansed surface.

4) Observations with SEM revealed that the cleansed surface showed wrinkle-like irregularities with many holes of a diameter ranging from a maximum of 1.5 μm to smaller, whereas the sandblasted surface showed irregularities like a scratch of various sizes running to all directions, which were larger than irregularities of the cleansed surface. Surface treatments generally caused an indistinct micro-feature of the surface with flat contrast of the overall view, but remarkable dissolution was observed after treatment with nitric acid both in the sandblasted and in the cleansed surfaces, and very small particles in the sandblasted surface were observed after treatment with potassium permanganate, and a vague oxide film in the cleansed surface was observed after being anodized with phosphoric acid.

5) Observations of the debonded surfaces revealed that 4-META/MMA-TBB resin cement appeared to be continuously forming new adhesion even during immersion in water.

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REFERENCES


共沈法により調整した ZnO-Al₂O₃ 系粉砕よりなるリン酸亜鉛セメント
——Ca と F 添加の影響——

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Ca と F を添加した ZnO-Al₂O₃ 系粉砕を共沈法により調製し、沈殿物および焼成物の組成と形態に与える影響を検討した。また、リン酸亜鉛セメントとして応用した場合、その歯科理工学的性質に与える両元素の添加の影響についても検討した。

沈殿物は Zn と Al を含む塩基性炭酸水和物であり、焼成することにより ZnO と ZnAl₂O₄ が生成した。また Ca と F を多量に配合したものは CaF₂ が生成した。焼成した粉末中の ZnO 粒子の大きさは Ca と F の添加により増大したが、ZnO と ZnAl₂O₄ 量はほとんど変化しなかった。これらの粉末を市販のリン酸亜鉛セメント液と練和した場合、そのセメント硬化体は両元素の添加により、圧縮強さが減少し硬化時間が短くなり、セメント表面の中和が促進された。ときに、Zn/Al 比が 5 で Ca, F を少量添加し、焼結一粉砕により調整した粉末の硬化体は 1% 乳酸および 1% ケン酸中での解離度が市販品よりも小さい値を示した。

銀配合型充填用グラスアイオノマーセメントの機械的性質と歯質への接着強さ

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銀配合型と従来型（それぞれ歯科練和用と手練和用の 2 種）を計 4 種の市販グラスアイオノマーセメントの圧縮強さ、ダイアメタル引張り強さ、曲げ強さ、無処理のエナメル質、象牙質に対するせん断接着力を測定した。銀配合型は、従来型に比較して試験のすべてにおいて統計的に劣っていた。接着強さについて従来型と比較すると、銀配合型は象牙質に対する接着性には大差がないが、エナメル質に対しては有意に低い値を示した。銀配合型と従来型について、器械練和用と手練和用とを比較すると、機械的性質と接着強さのすべてに統計的に有意な差は認められなかった。ダイアメタル引張り強さと曲げ強さについては、エナメル質、象牙質に対するせん断接着強さと統計的に有意な関係が認められたが、圧縮強さについてはみられなかった。

4-META/MMA-TBB レジンセメントとの接着を改善するための
歯科用金銀パラジウム合金の表面処理について

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4-META/MMA-TBB レジンセメントと歯科用金銀パラジウム合金の接着を改善するための金銀表面の簡便な処理法の比較を、金属同志および金属とエナメル質を接着させて行った。サンドブラストを行って鍛造後の酸化膜を除去し、リン酸で陽極処理を行った場合、37℃の水中浸漬初期から 3 カ月後まで十分な接着強さを保持していたが、金パラセラフを用いて鍛造後の酸化膜を除去し、リン酸で陽極処理を行った場合は、水中浸漬 3 カ月目で接着強さの著しい低下を生じた。サンドブラストのみ行ったものは、水中浸漬初期の接着強さは小さかったが、水中浸漬 3 カ月目になると接着強さは増加した。

4-META/MMA-TBB レジンセメントは、水中浸漬中においても常に新しい接着を形成している事が示唆された。