Effect of the Pressure applied to the Acid-etched Enamel on the Adhesive Strength of the Bonding Agent

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Received on March 10, 1986

Acid-etched enamel can be damaged by careless instrumentation during the application of bonding agents. In such a case, the adhesive strength of the resultant bonding agent may be impaired to a considerable degree, resulting in an unfavorable marginal integrity. The effects of applied pressure to the acid-etched enamel on the adhesive strength of the bonding agent were examined microstructurally. The influence of pressure was marked. For example, when a sponge pellet was used to apply the pressure, an adhesive strength of ca. 190 kg/cm² resulted following the applied pressure of 0.01-0.03 g. However, it decreased to ca. 100 and 50 kg/cm² when the applied pressure was 2-3 g and 7-10 g, respectively. Therefore, the bonding agent should be applied as carefully as possible.

Key words: Acid-etched enamel, Destruction, Bonding strength

INTRODUCTION

The acid-etching of enamel and subsequent application of a bonding agent are necessary procedures for the adhesion of restorations and appliances. Many investigations have been made on the acid-etching technique^{1,2)}, and it is now commonly used in clinical practice. However, even when acid-etching has made the enamel surface favourable for adhesion, factors inhibiting adhesion do exist³⁻⁵⁾, such as residual moisture of the etching solution, saliva, expiration, and destruction of the acid-etched enamel during the restorative procedure.

Previously we reported^{6,7)} that the destruction of the acid-etched enamel following the accidental contact of the instrument during the application of a bonding agent decreases the adhesive strength of the agent. However, in these studies, the pressure was not estimated quantitatively. In this study, we estimated preliminarily the pressure produced following the application of the bonding agent. Pressure was then applied to the acid-etched enamel, and the relationship between applied pressure and the adhesive strength of the bonding agent was examined. The effect of the instruments, such as sponge pellet, brush, cotton pellet, was examined morphologically and mechanically. The microleakage between the bonding agent and acid-etched enamel was also examined.

MATERIALS AND METHODS

1. Preparation of specimens

The labial surface of freshly extracted human permanent central incisor, stored in

distilled water, was flattened with sand paper (JIS # 600) and mounted in self-curing acrylic resin. Exposed enamel was etched with a 40 % aqueous solution of phosphoric acid, rinsed with water and dried. It was fixed to the table to an electronic balance with tacking tape. The instrument used for applying the bonding agent was fixed to the travelling stage of a microhardness tester. The tip of the instrument was put in contact with the acid-etched enamel and moved horizontally at a speed of 20 mm/min. This procedure was repeated by maintaining the distance of 0.5 mm.

Figure 1 shows the pressure applied. With a sponge pellet (Kuraray, included in the package of Clearfil F-II), at a pressure of 0.01-0.03 g, its original shape was maintained, at 0.2-0.6 g, its height was reduced to approximately half, and at 8-12 g it was flattened. With a brush (Tokuyama Soda, included in the package of Palfique), the pressure was 0.2-0.3 g, 2-4 g and 13-16 g when about 2, 4 and 6 mm of its tip was in contact, respectively. The cotton pellet had a similar impact as the sponge pellet.

Fig. 1 Pressures applied to the acid-etched enamel by a sponge pellet (top), brush (middle) and cotton pellet (bottom).

In addition to the above-mentioned specimens which had cross-sectioned enamel, specimens having longitudinally sectioned enamel were made. A premolar was used to make the specimen, whose crown was ground bucco-lingually.

2. Determination of adhesive strength

A polyethylene ring, 3.2 mm in diameter inside, was fixed on the acid-etched enamel surface with double-sided tack tape. The bonding agent (Kuraray, Clearfil New Bond) was applied carefully by its own weight, and a composite resin was then applied. Ten minutes later, the ring was removed. The resultant specimen was stored in water at 37°C for 24 hours.

The adhesive strength was determined by a testing machine with a cross-head speed of 2 mm/min. The testing procedure was described in detail, elsewhere⁸⁾. In each experiment, 9-12 determinations were made.

3. Examination of marginal leakage

A pressure of 7-13 g was applied to the acid-etched enamel with a cotton pellet, then, a thermal cycle (4 and 60°C, maintained 1 minute, alternatively) was applied 400 times and stained with fuchsin. A scanning electron micrograph was made. An acid-etched enamel surface, to which a pressure of 0.01-0.03 g was applied by a sponge pellet, was used as a control.

RESULTS AND DISCUSSION

Figure 2 shows the adhesive strength of bonding agent to the acid-etched enamel, to which the pressure was applied with a sponge pellet.

Fig. 2 Adhesive strength of bonding agent to the cross-sectioned, acid-etched enamel to which pressure was applied by a sponge pellet.

Figure 3 shows the SEM observations of the enamel surface. In these cases, the cross-sectioned enamel was exposed, and the adhesive strength was 185 kg/cm² when the pressure of 0.01-0.03 g was applied, but, it decreased with the increase in the applied pressure and at 7-10 g, the resultant adhesive strength decreased to as little as 50 kg/cm². As is apparent in Fig. 3, the etched enamel was damaged by the contact of the sponge pellet. Therefore, the greater the applied pressure, the more damaged the enamel thus showing smaller adhesive strength. The lack of an interlocking force following the insufficient formation of the resultant resin tag is apparent as shown in Fig. 4. As it is also apparent in

Fig. 3 SEM of enamel employed in Fig. 2.

Fig. 4 SEM of bonding agent (resin tag) resulting from Fig. 3.

Figs. 3 and 4, the acid-etched enamel was not damaged following the application of 0.01-0.03 g, indicating that the resultant resin tag is favourable for adhesion.

Figures 5, 6, 7 show the SEM observations on the adhesive strength of the bonding agent to the longitudinally sectioned acid-etched enamel. Adhesive strength of 110 kg/cm² is obtained following the pressure of 0.01-0.03 g. It decreased with the increase in applied pressure, and with the pressure of 7-10 g, the adhesive strength was as little as 40 kg/cm². This tendency is similar to that of the above mentioned cross-sectional enamel. The adhesive strength was 20-40 % lower than that of the cross-sectional enamel.

This is considered to be based upon the difference in mechanical properties and surface area of hydroxyapatite in acid-etched enamel, following its arrangement⁹⁾.

Figure 8 shows the adhesive strength of the bonding agent to the acid-etched cross-sectioned enamel, to which pressure was applied with a brush. Figure 9 shows the SEM observations. Unlike the case of the sponge pellet, a bonding strength of 60 kg/cm² was obtained following the large pressure of 13-16 g, which is not so different from the case of a small pressure of 2-3 g. This is considered to result from the reduction in the stress following

the expansion of the brush (Fig. 1) by the application of such a large pressure. This is indicated by the SEM observation in Fig. 9.

Figure 10 shows adhesive strength of a bonding agent to the acid-etched enamel, to which pressure was applied with a cotton pellet. Figure 11 shows a SEM observation when a pressure of 7-10 g was applied. A similar tendency as with the sponge pellet and brush was observed. However, following the application of a small pressure (0.01-0.03, 0.2-0.6 g), the resultant strength was 15-25 % lower than that of the sponge pellet. This is also considered

Fig. 5 Adhesive strength of bonding agent to the longitudinally sectioned, acid-etched enamel to which pressure was applied by a sponge pellet.

Fig. 6 SEM of enamel employed in Fig. 5.

Fig. 7 SEM of bonding agent (resin tag) resulting from Fig. 6.

Fig. 8 Adhesive strength of bonding agent to the cross-sectioned, acid-etched enamel to which pressure was applied by a brush.

to result from the difference in the stress during pressure application. The scratches are considerably larger than those produced with a sponge pellet.

In the microleakage test, no penetration of dyes between the bonding agent and acid-etched enamel was observed in the control (applied pressure is 0.01-0.03 g). However, the acid-etched enamel, to which a pressure of 7-13 g was applied, indicated penetration of the

Fig. 9 SEM of enamel employed in Fig. 8.

Fig. 10 Adhesive strength of bonding agent to the cross-sectioned, acid-etched enamel to which pressure was applied by a cotton pellet.

dyes, following microleakage. The SEM photograph in Fig. 12 show the interface between acid-etched enamel and bonding agent. When the applied pressure was small (0.01-0.02 g), the resin tag along the acid-etched enamel prism was regularly shaped. However, when a large pressure (7-13 g) was applied to the acid-etched enamel, the resultant resin tag was irregular in shape, indicating a lack of interlocking between acid-etched enamel and resin tag.

Fig. 11 SEM of enamel employed in Fig. 10 (pressure of 7-10 g was applied).

Fig. 12 SEM of the interface between acid-etched enamel and bonding agent (resin tag) after thermally cycled. Left, a small pressure (0.01-0.03 g, control); right, a large pressure (7-13 g) was applied.

Therefore, it is apparent that a pressure of only a few grams applied on the acid-etched enamel could impair the interlocking between bonding agent. This means that the strength of an acid-etched enamel is very small. Extreme care should be taken during the application of the bonding agent and adhesives. It is particularly important during the orthodontic and adhesive restorative procedures, because the surface of the acid-etched enamel is very large in these cases.

CONCLUSION

Various pressures were applied on the acid-etched enamel with several kind of instruments. The adhesive strength of the bonding agent to this surface was determined and a SEM observation was made. A microleakage test was also carried out. The following results were obtained :

1. The adhesive strength of the bonding agent was 185 kg/cm² when a pressure of 0.01-0.03 g was applied by a sponge pellet. It decreased with an increase in the pressure, and at 7-10 g, it became as small as 50 kg/cm². In the case of the longitudinally sectioned enamel,

the adhesive strength was 20-40 % smaller than that of the cross-sectioned.

2. When a brush or cotton pellet was used for the application of pressure, similar trends as described above were observed. Yet, the adhesive strength obtained was 15-25 % smaller than that using a sponge or brush, when a small pressure of 0.01-0.6 g was applied, using a cotton pellet.

3. When a large pressure, such as 7-13 g, was applied, microleakage occurred between the tooth structure and bonding agent.

4. An apparent relationship was observed between the microstructure of the acid-etched enamel and adhesive strength of the bonding agent.

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を SEM にて観察した。引っ張り試験を終えた試料を観察するとセメントは常にピンに付着しており、セメントとピンホルルの界面にて剥離を生じていた。引っ張り試験の結果、同じ直径のピンのグループでは保持面積が保持力に比例したが、ピンの直径が異なるとそれに対応し

たピンホルルの表面の粗さが異なり、そのために単位面積あたりの保持力が違ってきて保持面積が同じでも保持力が異なるという結果となった。

まとめ 1) 保持力は保持面積に比較する。

2) 表面粗さが保持力に大きく影響する。

歯科鑄造用合金とリン酸塩系埋没材の熱膨張に関する研究

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3種の市販リン酸塩系埋没材の練和時間と練和液を変えて、円柱状試片を作り、示差熱膨張計にて室温より900°C迄の熱膨張を測定した。また18種の歯科鑄造用合金をアルゴン雰囲気下にて融点より100~250°C低い温度迄の熱膨張を測定し、合金の融点における熱膨張値をGrüneisenの公式にもとづいて推定した。埋没材は硬化膨張が小さいと蠟型の変形を起こすことがないと言われ

ているが、硬化膨張が小さく900°Cにおける熱膨張が最大であったのは、Platoritの20%グリセリン水溶液60秒練和の場合で、熱膨張値は $1.31 \pm 0.02\%$ であった。埋没材と合金の熱膨張を比較すると前者のみで後者を十分に補償することができないのは明らかである。どの埋没材も硬化膨張を小さく抑えた場合、今回の練和条件下では鑄造体は小さくなるものと思われる。

構成元素の溶出量から検討したアマルガムの腐食

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一般型及び高銅型アマルガム構成元素の溶出量を測定し、 γ_1 相、 γ_2 相、 η 相の腐食比を求めた。

両アマルガムから溶出した銀に対する水銀量は、 γ_1 相の比よりも小さかった。また、一般型アマルガムからは多量の錫が溶出したが、 γ_2 相として対応するはずの水銀が全く検出されなかった。一方高銅型アマルガムからは、

η 相の比で銅と錫が溶出した。また腐食の要因は、 γ_1 相にとって時間と温度が、 γ_2 相には溶液と時間が、 η 相には全てが有意であった。

そして腐食後の微小組織の観察から、両アマルガムによる修復物の辺縁破折の機構の違いが示唆された。

酸エッチングエナメル質に加えられた圧が、

ボンディング材の接着強さに及ぼす影響

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ボンディング材を施す際の不注意によって酸エッチングされたエナメル質の破壊がおこることは充分予想される。このような場合には接着強さが著しく低下し、辺縁部の性状が劣化する可能性が大きい。本研究においては、酸エッチングされたエナメル質に加えられた圧がボン

ディング材の接着強さに及ぼす影響を機械的性質と微細構造の両面から検討した。圧の影響は非常に大きく、スポンジペレットを用いた場合、0.01~0.03gの微少な圧では約190 kg/cm²の接着強さが得られたが、圧が2~3g、7~10gと増大するに従って、それぞれ100 kg/

cm², 50 kg/cm²と著明に低下した。これらの事実から、酸エッチングされたエナメル質にボンディング材を施す

際には出来る限り慎重におこなうべきであることが確認された。

接着性レジンと合金の接着機構の解明に関する研究

—— ESCA によって解析された Co-Cr 合金の表面構造とレジンの接着強さの関係 ——

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70 mass %Co-30 %Cr 合金の 3 つの異なる表面状態 (バフ研磨したまま, 300°C と 500°C で大気中で酸化) に対する接着性レジン (4-META 添加) の接着性について検討した。合金の表面構造は ESCA と反射電子回析で解析し, 接着性と表面構造の関係について接着モデルを仮定して論じた。

接着性は, 研磨したままの方が 300°C と 500°C で酸化したものより優れていた。この接着性の優劣は, 液体チッ素を用いた熱サイクルを施すことによって見出された。

研磨したままの表面構造は, 20~30 Å の非晶質の不動態被膜で, Cr³⁺ や Co²⁺ または Co³⁺ を中心にして -OH や H₂O が 6 配位で結合していると考えられた。また, 300°C の場合は, 120 Å の酸化被膜が形成され, その表面は, 主に Co₃O₄ から成っている。酸化層の表面には数分子の H₂O が吸着していると考えられ, この吸着水の存在が, 研磨したままの表面よりも接着性を低下させた原因と考えられた。

表面滑沢硬化処理床用レジンの耐摩耗性

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本研究では, 表面滑沢硬化処理した床用レジンの耐摩耗性を表面粗さ R_{max}, R_z, R_a 及びブラッシング消失量の観点から検討した。すなわち, 表面滑沢硬化処理剤, Linkle Coat (Coating 1) 及び Permacure Link (Coating 2) の 2 種類を用いて, それら処理したものと無処理のものとを

とを統計的に比較, 検討した。その結果, 表面滑沢硬化処理した場合, その耐摩耗性は無処理のコントロールよりもより優れており, とくに, Coating 1 による床用レジンの消失量は摩耗回数の増加にもかかわらず Coating 2 のものよりも少ないことが判明した。

金属メッシュプレートの義歯床への応用

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床義歯装着時におこりうる床下粘膜組織にみられる弊害をできるだけ軽減し, 粘膜の生理機能を自然状態に保持できるような床用材料 TRUTISSU を試作し, いくつかの基礎実験を行なうとともにモニターによる臨床観察をも行ない, 以下のような評価を得た。1. TRUTISSU の成形性はよく, 成形後の適合性は良好であった。2. 口蓋粘膜に対する床の吸接着維持については他の材料と

くらべて同等の維持力が示された。3. 生物学的適合性については TRUTISSU の構成金属線に対して良好な細胞の接着ぬれを示し, 何らの変性も認められなかった。また, 口蓋粘膜をシミュレートした人工粘膜上の細胞について TRUTISSU 直下の細胞は正常な増殖を示した。4. コバルト・クロム床やレジン床との比較について, 装着感, 温度感および嗜好品に対する味覚などに