Contraction Gap versus Shear Bond Strength of Dentin Adhesive in Sound and Sclerotic Dentins

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To evaluate the effect of a dentin adhesive on sclerotic dentin, contraction gap width and shear bond strength were measured. Dentin cavity wall was pretreated with an experimental dentin bonding system with and without a dentin primer, or with a commercial dentin bonding system. In the experimental dentin bonding groups, contraction gap width of sclerotic dentin was significantly smaller than that of sound dentin when the cavity was not primed with glyceryl monomethacrylate. For each individual tooth, the correlation between contraction gap width and shear bond strength was insignificant. In conclusion, the bonding efficacy of dentin bonding systems to sclerotic dentin was superior to that of sound dentin. Further, it was determined that it was impossible to detect the interaction between the polymerization contraction stress of resin composites and the efficacy of dentin adhesives by measuring bond strength.

Key words: Contraction gap, Shear bond strength, Sclerotic dentin

INTRODUCTION

The bonding mechanism of dentin adhesives hinges on the hybrid layer formation on superficial dentin substrate. It was reported that bonding was obtained by the diffusion and polymerization of dentin adhesives in decalcified dentin\(^1\). In addition, the effect of priming on dentin bonding was explained by the expansion of microspaces throughout the dentin collagen network that has collapsed with acid etching. The monomer polymerizes after penetrating the enlarged collagen network, resulting in the hybrid layer formation. Therefore, the techniques and materials for forming the hybrid layer are essential to establishing durable dentin bonding\(^2\).

However, the primary requirement for a dentin bonding system is to prevent the separation of unpolymerized resin composite material from the dentin cavity wall during polymerization. The degree of polymerization contraction should be compensated by the flow of resin composite from the free surface into the cavity. From a clinical point of view, however, it is doubtful that the bond between resin composite and dentin cavity wall is obtained by monomer penetration into the dentin because a contraction gap is always formed at the adhesive interface. In addition, the structure inside the resin composite or dentin substrate has not been studied clinically, although they have been frequently observed in specimens for bond strength measurement. Thus, it may be speculated that marginal adaptation was established by the interaction between the dentin bonding agent and the physical characteristics of the dentin cavity wall. Most papers concerning dentin bonding systems evaluated the efficacy of dentin adhesives by measuring the bond strength, whereby bonding mechanism was then discussed based on the bond strength measured\(^3\). However, the relationship between the bond strength and cavity adaptation of resin composites has been questioned\(^4\).

Further, sound dentin has been employed as the substrate in most of the previous reports. Clinically, the dentin bonding system is frequently applied to sclerotic dentin that is physically and chemically degenerated. On this note, dentin adhesives have been reported to be less efficacious on sclerotic dentin than on sound dentin, because of inadequate monomer infiltration\(^5\).\(^-\)\(^8\). Moreover, acid-etching sclerotic dentin has been strongly recommended to ensure formation of the hybrid layer\(^9\). On the other hand, Tani and Kusunoki et al. demonstrated that the efficacy of dentin adhesives on sclerotic dentin was never inferior to that on sound dentin because a high monomer concentration was maintained at the adhesive interface\(^9\).\(^10\).

The purpose of this study was to examine the efficacy of dentin adhesives on sound and sclerotic dentins and to clarify the relationship between contraction gap width and shear bond strength.

MATERIALS AND METHODS

Contraction gap measurement
Thirty extracted sound human teeth and thirty extracted human molars with sclerotic dentin were used. Sclerotic dentin was diagnosed visually as having definite brown discoloration with high translucency. The efficacy of a commercial dentin bonding
system and that of an experimental dentin bonding system on sound and sclerotic dentins were evaluated by measuring the contraction gap width and shear bond strength.

Proximal enamel of the extracted human tooth was removed and flattened using 220-grit wet silicon carbide paper, and a cylindrical cavity — approximately 3 mm in diameter and 1.5 mm in depth — was prepared in the exposed dentin using the end of a plain fissure bur (Figs. 1(1), (2), (3)). Dentin cavity wall was treated with a commercial dentin bonding system (Clearfil Mega Bond, Kuraray Medical, Tokyo, Japan) according to manufacturer’s instructions. It is one of the self-etching systems that contains a non-rinse conditioner which may be classified as a mild conditioner. In this way, we sought to prevent as much as possible the decalcification of dentin beneath the smear layer.

The cavity was then slightly overfilled with a commercial resin composite (Silux Plus, 3M, MN, USA). Surface of the resin composite was covered by a plastic matrix, and a glass plate gently pressed on it momentarily. The resin composite was polymerized by irradiation for 40 seconds using a commercial lamp unit (White Light, Morita, Tokyo, Japan). After placing the specimens in tap water at room temperature (24 ± 1°C) for 10 minutes, surplus resin composite was eliminated using 1500-grit wet silicon carbide paper. Exposed cavity margin was then polished with a linen cloth mediated with alumina slurry with a grain size of 0.03 μm (Fig. 1(4)).

Marginal integrity was observed under a light microscope (Orthoplane, Leitz, Wetzlar, West Germany), and the width of possible contraction gap was measured using a screw micrometer (Eypeiece Digital, Leitz, Wetzlar, West Germany) mounted on an ocular lens of the microscope. Contraction gap width was measured at eight points every 45 degrees along the cavity margin at 1024 × magnification (Fig. 1(5)). Contraction gap value was calculated as the sum of diametrically opposing gap widths as a percentage of the cavity diameter. The maximum of the four gap values was then assigned as the maximum contraction gap value of the specimen.

For the positive control group, the dentin cavity wall was conditioned with 0.5 mol/L neutralized ethylene diamine tetraacetic acid (EDTA, Dojin, Wako Pure Chemical Industries Ltd., Osaka, Japan) (pH 7.4) for 60 seconds, followed by rinsing and drying. The cavity was then primed with 35 vol% glyceryl mono-methacrylate (Blemmer GLM, NOF Corp., Tokyo, Japan) (GM) solution for 60 seconds, followed by air blasting without rinsing. A commercial dual-cured dentin bonding agent (Clearfil Photo Bond, Kuraray Medical, Tokyo, Japan) was applied to the cavity and irradiated for 10 seconds after eliminating the excess material using a gentle air blast. Placing of light-activated commercial resin composite filling was performed, and gap width was measured using the same method as that for the commercial dentin bonding system group mentioned above. In another control group, the cavity was not primed with GM solution, although other steps as per those for the positive control group were carried

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Fig. 1 Contraction gap measurement method.

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(1) Extracted human molar  (2) Flattening a proximal surface  (3) Cylindrical cavity preparation
(3 mm in diameter and 1.5 mm deep)

(4) Resin composite filling mediated with dentin adhesive  (5) Contraction gap width measurement under a light microscope
out. Ten specimens for each group, sixty in total, were prepared.

Shear bond strength measurement
After contraction gap width was measured, the specimen was embedded in an epoxy resin (Epofix, Struers, Copenhagen, Denmark). The opposite proximal enamel was removed, and a flat dentin surface prepared using 600-grit wet silicon carbide paper (Fig. 2(1)). A split Teflon ring - 3.6-mm inner diameter, 20-mm outer diameter, and 5-mm height - was clamped onto the exposed flat dentin surface. Then, the resin composite was placed on the dentin in the cavity of the Teflon mold after dentin was pretreated by one of the abovementioned three bonding systems. The resin composite, with a thickness of approximately 2 mm, was irradiated for 40 seconds from the top of the center hole of the Teflon mold (Figs. 2(2), (3)). Immediately after irradiation, the specimen was placed in tap water at room temperature (24±1°C) for 10 minutes, and shear bond strength was measured using a universal testing machine (Instron 4302, Mass, USA) at a crosshead speed of 5 mm/min (Fig. 2(4)).

SEM observation of dentin surfaces and adhesive interfaces
Structure of the sclerotic dentin after EDTA conditioning was observed using a scanning electron microscope (S-4700, Hitachi, Tokyo, Japan), and likewise the marginal adaptation after treating with 1 N hydrochloric acid for 20 seconds following measurement of contraction gap width. The specimens were dehydrated in a gradual ethanol solution - in which the concentration was increased from 70% to 95% (70%, 80%, 90%, 95%) - for 30 minutes, and then in 99% for two 15-minute periods. After which, specimens were critical point dried and sputter-coated with palladium and platinum.

RESULTS

Contraction gap measurement
Contraction gap measurements are shown in Table 1

(1) Embedding the specimen in an epoxy resin and flattening the opposite proximal surface

(2) Rein composite filling in the Teflon mold mediated with the dentin adhesive

(3) Specimen for the shear bond strength measurement

(4) Shear bond strength measurement

Fig. 2 Shear bond strength measurement method.

Table 1 Contraction gap widths measured in this study

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<th>EDTA, GM, CPB</th>
<th>EDTA, CPB</th>
<th>MEGA Bond</th>
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<td>Sclerotic dentin</td>
<td>0 (10)</td>
<td>0.023±0.029 (4)*</td>
<td>0.021±0.015 (3)*</td>
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<tr>
<td>Sound dentin</td>
<td>0 (10)</td>
<td>0.091±0.061 (1)</td>
<td>0.040±0.048 (4)*</td>
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CPB: Clearfil Photo Bond.
N=10; Mean ± SD of marginal gap width as a percentage of cavity diameter.
( ) : Number of gap-free specimens out of 10 specimens.
*: Difference is not statistically significant (Kruska-Wallis test, p<0.05).
Values with vertical line are not significantly different statistically (Mann-Whitney U test, p<0.05).
and Fig. 3. Formation of a contraction gap was prevented completely in the positive control group, regardless of the kind of dentin substrate. When the cavity was not primed in the experimental dentin bonding system, the gap value of sound dentin was significantly greater than that of sclerotic dentin, according to statistical analysis using the Mann-Whitney U test (p<0.05). With the commercial dentin bonding system, gaps were observed in more than half of the specimens, although the difference between sound and sclerotic dentins was insignificant. Further, between the two commercial dentin bonding groups and the non-primed experimental sclerotic dentin group, differences were not statistically significant according to Kruskal-Wallis one-way analysis of variance by ranks (p>0.05).

**Shear bond strength measurement**

Shear bond strength measurements are shown in Table 2 and Fig.3. The shear bond strength measured was not significantly affected by the dentin condition in the three dentin bonding systems tested (Student’s t-test, p>0.05). Moreover, correlation between contraction gap value and shear bond strength was insignificant (Fig. 4) with R^2 (correlation coefficient) = 0.009.

**SEM observation of dentin surfaces and adhesive interfaces**

With EDTA conditioning, the smear layer was completely removed. Sound dentin surface conditioned with EDTA is shown in Fig. 5(a), where dentinal tubules were enlarged. Sclerotic dentin surface conditioned with EDTA is shown in Fig. 5(b), where dentinal tubules were closed.

Complete marginal adaptation was obtained regardless of the kind of dentin substrate when primed with the GM solution using the experimental dentin bonding system. It should be mentioned that it was difficult to distinguish a bonding layer from a hybrid layer when the experimental dentin bonding system was compared with the commercial dentin bonding system. This was because the hybrid layer formation and bonding layer were too thin to be observed. However, sclerotic dentin differed from normal dentin in that resin tags were not observed (Figs. 6(a), (b)).

Contraction gap between resin composite and

<table>
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<th>Table 2 Shear bond strengths measured in this study</th>
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<td>EDTA, GM, CPB</td>
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<tr>
<td>Sclerotic dentin</td>
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<td>Sound dentin</td>
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CPB: Clearfil Photo Bond.
N=10; Mean ± SD of shear bond strength (MPa).
Values with dotted line are significantly different statistically (one-way ANOVA Fisher’s PLSD, p<0.05).

**Fig. 3** Results of contraction gap widths and shear bond strengths.

**Fig. 4** Correlation and linear regression between the contraction gap width in the cylindrical dentin cavity and the shear bond strength of resin composite to the flat dentin surface.
sound dentin when experimental dentin bonding system was used without GM priming is shown in Fig. 7(a). Adhesive interface between resin composite and sclerotic dentin when experimental dentin bonding system was used without GM priming is shown in Fig. 7(b). Complete marginal integrity was obtained in four out of 10 specimens— that is, marginal integrity was greater compared to that of sound dentin.

Adhesive interface between resin composite and sound dentin when used with commercial dentin bonding system is shown in Fig. 8(a). The hybrid layer was not distinct and the bonding layer was thick. Resin tags were observed in some dentinal tubules. In parallel, adhesive interface between resin composite and sclerotic dentin when used with commercial dentin bonding system is shown in Fig. 8(b).

![Sound dentin](image1)

**Fig. 5**

a. Sound dentin surface conditioned with EDTA for 60 sec. Smear layer is completely removed and dentinal tubules are enlarged.
b. Sclerotic dentin surface conditioned with EDTA for 60 sec. Dentinal tubules are closed by deposits of an inorganic component.

![Sclerotic dentin](image2)

![Sound dentin](image3)

![Sclerotic dentin](image4)

**Fig. 6**

a. Complete marginal adaptation between resin composite and sound dentin when experimental dentin bonding system is used with GM priming. Hybrid layer formation and bonding layer are too thin to be observed. D: Dentin; RC: Resin composite.
b. Complete marginal adaptation between resin composite and sclerotic dentin when experimental dentin bonding system is used with GM priming. Hybrid layer formation, bonding layer, and resin tag formation are not distinct. D: Dentin; RC: Resin composite.
DISCUSSION

In 1982, Nakabayashi proposed that the bonding mechanism of dentin bonding systems could be explained by a hybrid layer formation in the superficial dentin layer\(^1\). In addition, as demonstrated by Sugizaki, Gwinett and Van Meerbeek \textit{et al.}, dentin priming as well as a moist dentin were considered effective and essential for expanding the microspaces throughout the collagen network in which the monomers penetrated and formed the hybrid layer\(^2,12-15\).

However, the detailed mechanism of dentin bonding by hybrid layer formation was based on bond strength measurement and scanning electron microscopic observations of the microstructure at the adhesive interface. It was impossible to detect—through these evaluation methods—the effect of the polymerization contraction stress of resin composites on marginal integrity because resin composites shrank toward the flat dentin surface during polymerization.

Asmussen claimed that the bonding efficacy of dentin adhesives should be examined in a cylindrical
dentin cavity, because marginal integrity was to be determined by the interaction between the bonding efficacy of dentin adhesives and the polymerization contraction stress of resin composites. Furthermore, Feilzer et al. reported that the proportions of the dentin cavity wall and the surface free of resin composite restoration significantly influenced cavity adaptation. In addition, Wu et al. demonstrated that the contraction gap width of resin composites increased in proportion to the surface free of the resin composite restoration and to that of the dentin cavity wall. These reports suggested that the efficacy of dentin adhesives should be evaluated consistently not by bond strength measurement, but by observation of marginal integrity. As demonstrated in this study, the correlation between contraction gap width and shear bond strength of individual teeth was quite insignificant. Therefore, the clinical performance of a dentin bonding system should be estimated by the contraction gap value rather than by shear bond strength value.

In this study, the marginal integrity of resin composites in cylindrical dentin cavities was examined according to the contraction gap width measurement method reported by Asmussen. Contraction gap formation was prevented by a possible interaction between the functional monomer and the inorganic component of dentin. This was because gap width increased as dentin hardness was reduced by dentin conditioning. In addition, intermediate resin without any functional monomer such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) or 4-methacryloyloxyethyl trimellitate anhydride (4-META) could not prevent contraction gap formation. The effect of GM priming has been explained by the physical prevention of both fluid flow through the dentinal tubules and monomer diffusion into the dentin. Thus, GM priming ensured polymerization of the chemical compound of the functional monomer and the inorganic component of dentin substrate at the adhesive interface.

As mentioned above, dentin bonding mechanism has been widely explained by monomer diffusion into the dentin collagen network. In addition, it was commonly believed that monomer penetration into sclerotic dentin was limited as compared to that into sound dentin. Therefore, the bond strength of dentin adhesives to sclerotic dentin has been frequently reported as being lower when compared against sound dentin. To promote monomer infiltration, it was recommended to etch the sclerotic dentin more strongly.

As shown in this study, however, gap width was smaller in sclerotic dentin cavities versus the sound dentin cavities when the dentin cavity wall was not primed with GM solution. Such a difference might be caused by the limited infiltration of the chemical substances into the dentin substrate. Adhesive monomer diffusion into sclerotic dentin was interrupted, and a high monomer concentration was maintained at the adhesive surface. In addition, water penetrating the dentinal tubules was also restricted, thereby ensuring monomer polymerization. The limited monomer diffusion in the sclerotic dentin was further disturbed by GM priming, and hence resulting in complete marginal adaptation of the resin composite restoration. From a clinical point of view, sclerotic dentin should neither be removed nor etched by dentin conditioner because a high monomer concentration should be maintained at the adhesive interface. In addition, GM priming should be recommended regardless of the kind of dentin substrate to avoid contamination of the adhesive surface by water and to prevent adhesive monomer diffusing into the dentin.

As discussed above, it was concluded that the bonding efficacy of dentin adhesives should be evaluated by contraction gap width measurement. In addition, limited monomer diffusion into sclerotic dentin was considered desirable for bonding because a high monomer concentration was maintained at the adhesive interface coupled with negligible contamination to the cavity wall by water.

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