

Micro-shear bond strength of resin-modified glass ionomer and resin-based adhesives to dentin

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국문초록

상아질 위치에 따른 접착성 수복재의 미세전단결합강도에 관한 연구

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이 연구의 목적은 수종 상아질 접착 시스템과 복합 레진 및 resin-modified glass ionomer를 상아질 표면에 접착하여 재료 및 상아질내 위치에 따른 미세전단결합강도를 측정, 비교하는 것이다.

상아질 접착 시스템으로는, 3-step인 Scotchbond Multi-Purpose Plus, 2-step인 Single Bond와 자가 부식형 시스템인 Clearfil SE Bond를, 1-step인 Prompt L-Pop을 사용하였다. 이와 함께 hybrid type의 복합 레진인 Clearfil AP-X와 Z250을 사용하였으며 resin-modified glass ionomer로는 Fuji II LC를 사용하였다. 상악 소구치를 치아의 근원심 중앙부를 절단하여 상아질면을 노출시켰다. 5개 실험군으로 분류하고 상아질면을 위치에 따라 치관부의 occlusal 1/3, middle 1/3, cervical 1/3과 치근부로 구분지어 시편을 부착하였다. 미세전단결합강도측정은 Universal testing machine(EZ-test; Shimadzu, Japan)에서 측정하였다.

Occlusal 1/3부위에서는 SE가 가장 높은 값을, SM과 SB간에는 유의차가 없었으며, PL, GI순으로, Middle 1/3부위에서는 SM \geq SE \geq SB \geq PL \geq GI 순으로, cervical 1/3부위에서는 SM, SE, SB간에 유의차가 없었다. Root dentin에서는 SM이 가장 높은 값을 보였으며 SE, PL, GI간에 유의차가 없었다. SE만이 치관부 상아질에 비해 치근부에서 유의할만한 결합강도의 감소를 나타냈다($p < 0.05$). GI는 치관부 상아질에서는 다른 군에 비해 유의성 있게 낮은 결합강도를 보였으나 치근부에서는 SE, PL과 유의차가 없었다.

주요어 : 접착성 수복재, 미세전단결합강도, Resin-modified glass ionomer 치관부 상아질, 치근부 상아질, 상아질 접착 시스템

I. INTRODUCTION

Dentin is a complex biological structure. It consists of highly oriented microstructure dominated by tubules. This structure leads to variation in tubular size, number and content, quantity of intertubular dentin¹⁾. As previous studies, the various structural components and properties of dentin can directly affect the adhesive bonding to tooth tissues^{2-5,9)}. Biological and clinical factors such as dentin permeability, pulpal fluid flow,

sclerosis and dental caries can also affect dentin bonding⁶⁾. There is little evidence for the chemical bonding. But, bonding to dentin can be accomplished by mechanical retention provided by resin tags and hybrid layer formation. It is so called, micromechanical interlocking as the resin forms taglike extensions into the etched enamel surface. However, dentin is a less preferable substrate than enamel for resin bonding. Many factors contribute to this situation : high organic contents, pulpal fluid flow, odontoblastic process, smear

layer and so on. The achievement of the bond between adhesive resin and dentin depends on the penetration of the primer and adhesive resin into the conditioned dentin surface in order to create micromechanical interlocking between the collagen in the dentin and resin, to form a hybrid layer or resin-dentin interdiffusion zone^{11,13)}. The penetration of primer and adhesive resin into dentin may be affected by regional difference and the properties of dentin substrate. Researches have shown that the bond strengths to deep dentin were lower than those to superficial dentin^{4,14,15)}. Some studies have reported that bond strengths to crown and root dentin varied according to the adhesive materials and site in the dentin^{2,16,17)}.

A hybrid layer can be produced by etching and priming of the dentin followed by applying adhesive resin to the dentin surface. Etching is necessary to remove the smear layer and to expose the collagen fibers in the dentin. During priming procedure, hydrophilic monomers diffuse through the demineralized dentin and displace water with polymerizable monomers and stabilize the hydrated collagen network. Next, adhesive resin is applied to the primed dentin and polymerized. Recently, according to the composition and chemistry of the materials, current materials can be classified as conventional adhesive system(three-step), self-priming adhesive system(two-step), self-etching adhesive system(two-step) and all-in-one system (one-step).

Most conventional adhesive systems (three-step) usually can produce high bond strength to dentin. But excessive etching produces weak bonding because collagen fibers at the base of the demineralized dentin are not completely impregnated by the resin. In addition, there is a risk of collagen collapse during air drying after etching^{18,19)}. Recently, self-priming adhesive system was introduced to simplify bonding procedure. Manufacturers have claimed that self-priming adhesives completely infiltrate the etched dentin with resin in one step. However, some recent studies have suggested that this combined system may reduce effectiveness of hybridization^{4,16,20,21)}.

Another approach is the use of self-etching adhesive system(two-step). In this system, etching and priming of the dentin occur simultaneously by infiltrating the smear layer-covered dentin with acidic resin. So, separate acid etching and rinsing steps are eliminated. Furthermore, the risk for incomplete impregnation of the demineralized dentin by the adhesive resin is avoided^{16,22)}. It was demonstrated in a previous nano-leakage study that compared self-etching systems with total-etching systems¹²⁾. However, it is still unclear if these materials could produce strong, durable bonds. All-in-one system(one-step) can be classified into a kind of self-etching adhesive system. Absolutely, it is a self-etching/self-priming adhesive system. It has been developed by raising the concentration of the acidic adhesive monomers from their original 5-6% concentration in conventional bonding systems to 20% or more.

Glass-ionomer is one of the two major groups of direct tooth-colored restorative materials. These materials have significantly different properties and characteristics, so can be divided some categories. Many attempts have been tried to make materials which have advantages of the composites and glass-ionomers. The first materials were produced for lining and base. Afterwards, variable materials have developed for restorations, core build up, luting, lining and base. Resin-modified glass ionomers have some characteristics of glass-ionomers and it is modified by the presence of resin. That is, these materials are setting through an acid-base reaction and polymerization. Bond strengths of resin-modified glass ionomers to the tooth tissues greater than those of conventional materials. And the bonding mechanism is not simple as for conventional glass ionomers. Bond strength to superficial dentin is stronger than to deep dentin such as the most dentin bonding systems. However, there is little evidence to support formation of hybrid layer for resin-modified glass ionomers²⁵⁾.

The direct bonded restorative materials allow the more conservative cavity preparation, and the remaining tooth structure may be reinforced by dentin bonding mechanism of these materials.

There are a variety of bond strength test methods to measure the bonding quality of adhesive systems^{27,28)}. Sano et al.²⁹⁾ have developed a micro-tensile bond test, which needs very small cross-sectional areas of dentin-resin specimen and leads a uniform stress distribution, so that most bond failures occur interfacially (adhesive failure). But during the preparation of dumbbell or hourglass-shaped specimens, stresses can be produced by cutting with high speed bur, and specimen itself can be fractured. Afterwards, Shono et al.³⁰⁾ reported a new version of the micro-tensile bond test. That is 'non-trimming' technique. They did not use a high speed bur but only used a low speed diamond saw during the preparation of specimens. But, this is not a simple procedure. In this study, bond strengths were measured by means of micro-shear bond test. This testing method has some advantages of micro-tensile bond test. And little stresses produce during the preparation of specimens comparing to the micro-tensile bond test. It is also a relatively simple method.

This study was designed to compare the micro-shear bond strengths of resin-modified glass ionomer and some kinds of resin-based adhesives according to the location in the dentin.

II. MATERIALS AND METHODS

Seventy five human upper premolars, which had been stored at 4°C in distilled water, were used in this study after removal of dental calculus and soft tissues. The teeth were embedded in the center of acrylic ring(diameter : 20mm, height : 15mm) with self-curing epoxy resin and mounted in a cut-off assembly of low speed diamond saw(ISOMET, Buehler, USA) for sectioning. The teeth were cut off vertically through the middle of the mesio-distal dimension, forming two halves. Then, the exposed flat dentinal surface was finished with 600-grit silicon carbide paper under running water to create standardized smear layer. One hundred fifty sectioned teeth were randomly divided into five groups : thirty sections for each experimental group(Fig. 1)

1. Specimen preparation

Four dentin bonding systems with two resin composites and one resin-modified glass ionomer were used in this study(Table 1, 2). Seven specimens were prepared in each exposed dentin surface according to the location. The location was divided to four areas : occlusal $\frac{1}{3}$, middle $\frac{1}{3}$, cervical $\frac{1}{3}$ of coronal dentin and root dentin.

For the conventional adhesive system (three-step), Scotchbond Multi- Purpose Plus(3M Dental Products, MN, USA), the dentin surface was acid-etched for 15seconds with the 37% phosphoric acid gel, rinsed for 10seconds and dried briefly to keep the dentinal surfaces visibly moist. Then, the primer was applied to the etched dentin and gently air dried. Finally the adhesive resin was applied and light-cured for 10seconds. Composite buildup of Z250(3M Dental Products, MN, USA) resin composite was performed with a tygon tube(SAINT-GOBAIN Performance Plastic Co., USA. inner diameter : 0.8mm, height : 1.0mm) and polymerized for 40seconds. For the self-priming adhesive system(two-step), Single Bond(3M Dental Products, MN, USA), the dentin surface was acid-etched for 15seconds with the 37% phosphoric acid gel, rinsed for 10seconds and dried briefly to keep the dentinal surfaces visibly moist. Then, the self-priming adhesive was applied two successive coats onto the etched surface and evaporated the excess of solvent with



Fig. 1. Prepared tooth specimen

Table 1. Dentin bonding systems used in this study

Materials(code)	Component	Composition	Manufacturer
Scotchbond Multi-Purpose Plus (SBMP)	Etchant	35% Phosphoric acid	3M Dental Products, St. Paul, MN, USA
	Primer	HEMA, water,	
	Adhesive	Copolymer of polyalkenoic acid HEMA, Bis-GMA	
Single Bond (SB)	Etchant	35% Phosphoric acid	3M Dental Products, St. Paul, MN, USA
	Adhesive	HEMA, Bis-GMA, Copolymer of polyalkenoic acid, ethanol, water	
Clearfil SE Bond (SE)	Primer	10-MDP, HEMA, water	Kuraray Co., Osaka, Japan
	Adhesive	10-MDP, HEMA, dimethacrylate microfiller	
Prompt L-Pop (PL)	Liquid 1 (red)	methacrylated phosphates, initiators, stabilizer	ESPE, Seefeld, Germany
	Liquid 2 (yellow)	water, fluoride complex, stabilizer	

Bis-GMA=Bisphenol-A glycidyl methacrylate

HEMA=Hydroxyethylmethacrylate

10-MDP=10-methacryloyloxydecyl dihydrogen phosphate

Table 2. Resin Composite and resin-modified glass ionomer used in this study

Material	Composition	Manufacturer
Clearfil AP-X	hybrid resin composite	Kuraray Co., Osaka, Japan
Z250	hybrid resin composite	3M Dental Products, St. Paul, MN, USA
Fuji II LC	fluoroaluminum silicate glass, polyacrylic acid, HEMA	GC Co., Tokyo, Japan

2~5seconds air blast. Then, the adhesive layer was light-cured for 10seconds. Composite buildup of Z250 resin composite was performed as previously described. For the self-etching adhesive system(two-step), Clearfil SE Bond (Kuraray Co., Osaka, Japan), the dentin surfaces were primed by self-etching primer and waited for 30seconds. After evaporating the solvent, the adhesive resin was applied and thinned with gentle air and light-cured 10seconds. Composite buildup of Clearfil AP-X(Kuraray Co., Osaka, Japan) resin

composite was performed as previously described. For the all-in-one system(one-step), Prompt L-Pop(ESPE, Seefeld, Germany), it was supplied in a patented disposable blister pack that consists of two pre-dosed compartments that might be connected via pressure application. This enabled the different components from the cushions to be mixed and activated immediately prior to use. The adhesive was brushed for 15seconds using the disposable applicator and thinned with air stream gently. And then light cured for 10sec-

onds. If the surface was not shiny, the repeated application was needed. Composite buildups of Z250(3M Dental Products, MN, USA) resin composite was performed as previously described.

In the resin-modified glass ionomer, Fuji II LC (GC Co., Tokyo, Japan), the 10% polyacrylic acid dentin conditioner was applied for 20seconds and rinsed for 10seconds and gently air-dried. Fuji II LC was applied as the procedure recommended by the manufactures.

After bonding procedures, all specimens were stored in distilled water at 37℃, for 24 hours prior to testing.

2. Micro-shear bond test

After storage of all specimens, the micro-shear bond test was performed. Each specimen was

placed in a testing machine(EZ-test : Shimadzu, Japan) for micro-shear bond testing. First, the specimen was engaged with the shear jig(Fig. 2, 3) And, a thin wire was looped around the composite resin cylinder, making contact through half of its circumference. A shear force was applied to each specimen at a cross-head speed of 1mm/min until failure occurred. The center of resin cylinder were aligned with the wire, the center of the load cell as straight as possible. The load at failure and the surface area for each specimen were used to calculate the bond strength in MPa.

3. Statistical analysis

Overall means and standard deviations (S.D.) of the micro-shear bond strength were calculated for each region; occlusal $\frac{1}{3}$, middle $\frac{1}{3}$, cervical $\frac{1}{3}$ of

Table 3. Code of five experimental groups by used materials.

Code	Materials	
SM	Scotchbond Multi-Purpose Plus	Z250
SB	Single Bond	Z250
SE	Clearfil SE Bond	Clearfil AP-X
PL	Prompt L-Pop	Z250
GI	Dentin Conditioner	Fuji II LC



Fig. 2. Universal testing machine

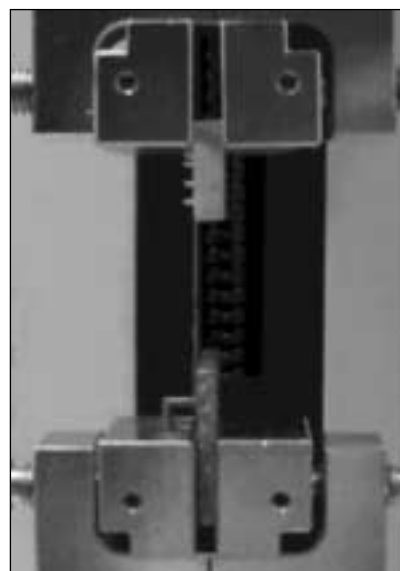


Fig. 3. Test mode with engaged specimen

coronal dentin and root dentin. The data were statistically analyzed using one-way analysis of variance (ANOVA) and Tukey's Multiple Comparison test at confidence level of 95% ($p < 0.05$).

III. RESULTS

1. Micro-shear bond strength

The means and standard deviations of the micro-shear bond strengths of the tested materials of five experimental groups are presented in Table 4 (Fig. 8).

For Scotchbond Multi-Purpose Plus (SM), there was no significant regional difference according to the location of dentin (Occlusal $\frac{1}{3}$ = 34.08 ± 5.13 MPa, Middle $\frac{1}{3}$ = 39.16 ± 6.96 MPa, Cervical $\frac{1}{3}$ = 35.03 ± 4.70 MPa, Root dentin = 34.27 MPa ± 7.36 ; $p > 0.05$). Also, Single Bond (SB) showed no significant difference according to the location of dentin (Occlusal $\frac{1}{3}$ = 32.08 ± 7.22 MPa, Middle $\frac{1}{3}$ = 31.81 ± 8.28 MPa, Cervical $\frac{1}{3}$ = 32.95 ± 7.94 MPa, Root dentin = 28.55 ± 7.56 MPa ; $p > 0.05$). Then, Prompt L-Pop (PL) showed the similar pattern of bond strength (Occlusal $\frac{1}{3}$ = 28.30 ± 8.72 MPa, Middle $\frac{1}{3}$ = 24.75 ± 8.95 MPa, Cervical $\frac{1}{3}$ = 26.12 ± 7.84 MPa, Root dentin = 22.39 ± 7.39 MPa ; $p > 0.05$). In this group, all the bond strengths were lower than those of other dentin bonding systems at the same location of dentin surface. Clearfil SE Bond (SE), the bond strength in the root dentin was significantly lower than those of other locations (Occlusal $\frac{1}{3}$ = 38.18 ± 7.80 MPa, Middle $\frac{1}{3}$ = 34.40 ± 7.82 MPa, Cervical $\frac{1}{3}$ = 34.48 ± 7.54 MPa, Root dentin = 22.58 ± 8.12 MPa ; $p < 0.05$). In the middle $\frac{1}{3}$, bond strengths

7.36 ; $p > 0.05$). Also, Single Bond (SB) showed no significant difference according to the location of dentin (Occlusal $\frac{1}{3}$ = 32.08 ± 7.22 MPa, Middle $\frac{1}{3}$ = 31.81 ± 8.28 MPa, Cervical $\frac{1}{3}$ = 32.95 ± 7.94 MPa, Root dentin = 28.55 ± 7.56 MPa ; $p > 0.05$). Then, Prompt L-Pop (PL) showed the similar pattern of bond strength (Occlusal $\frac{1}{3}$ = 28.30 ± 8.72 MPa, Middle $\frac{1}{3}$ = 24.75 ± 8.95 MPa, Cervical $\frac{1}{3}$ = 26.12 ± 7.84 MPa, Root dentin = 22.39 ± 7.39 MPa ; $p > 0.05$). In this group, all the bond strengths were lower than those of other dentin bonding systems at the same location of dentin surface. Clearfil SE Bond (SE), the bond strength in the root dentin was significantly lower than those of other locations (Occlusal $\frac{1}{3}$ = 38.18 ± 7.80 MPa, Middle $\frac{1}{3}$ = 34.40 ± 7.82 MPa, Cervical $\frac{1}{3}$ = 34.48 ± 7.54 MPa, Root dentin = 22.58 ± 8.12 MPa ; $p < 0.05$). In the middle $\frac{1}{3}$, bond strengths

Table 4. Micro-shear bond strength of experimental groups (unit : MPa \pm SD)

	Occlusal	Middle	Cervical	Root
SM	34.08 ± 5.13^a	39.16 ± 6.96^a	35.03 ± 4.70^a	34.27 ± 7.36
SB	32.08 ± 7.22^a	31.81 ± 8.28^b	32.95 ± 7.94^a	28.55 ± 7.56^a
SE	38.18 ± 7.80^A	$34.40 \pm 7.82^{a,b,A}$	34.48 ± 7.54^{A}	$22.58 \pm 8.12^{a,b,B}$
PL	28.30 ± 8.72	24.75 ± 8.95^c	26.12 ± 7.84	$22.39 \pm 7.39^{a,b}$
GI	19.48 ± 4.00	18.95 ± 5.49^c	19.83 ± 5.24	17.40 ± 4.65^b

Mean values with the same uppercase and lowercase superscript letters are not statistically different ($p > 0.05$).

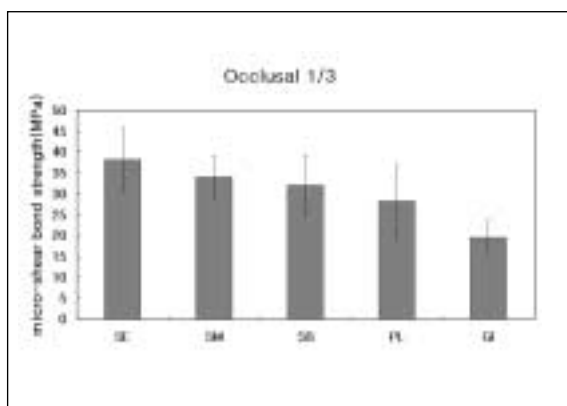


Fig. 4. Micro-shear Bond Strengths: Occlusal $\frac{1}{3}$ of coronal dentin

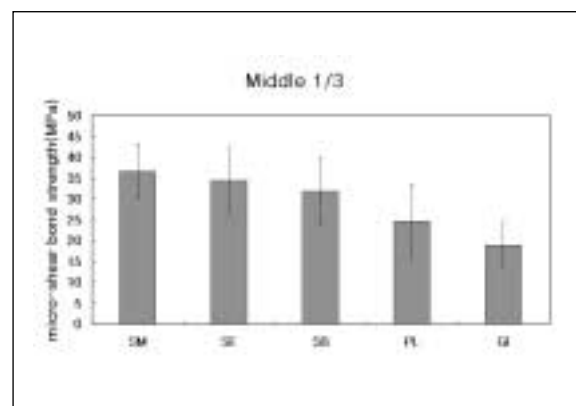


Fig. 5. Micro-shear Bond Strength: Middle $\frac{1}{3}$ of coronal dentin

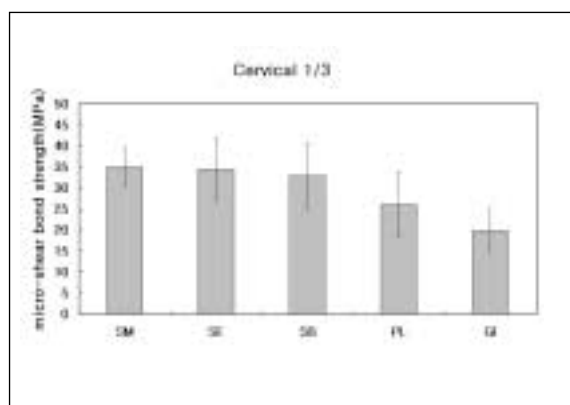


Fig. 6. Micro-shear Bond Strengths: Cervical $\frac{1}{3}$ of coronal dentin

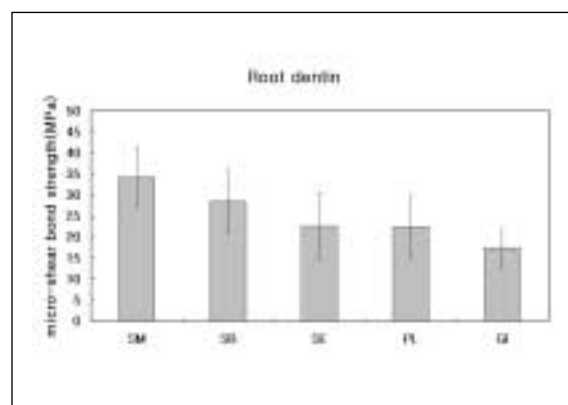


Fig. 7. Micro-shear Bond Strengths: Root dentin

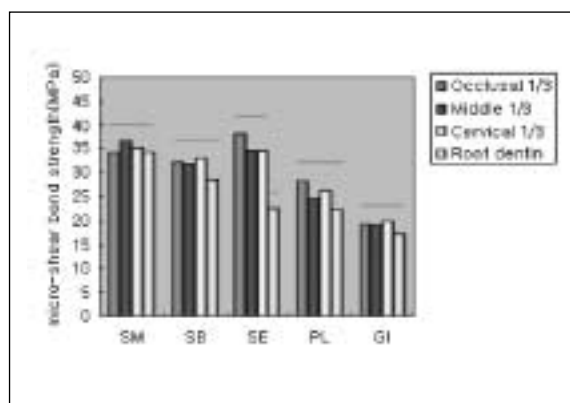


Fig. 8. Micro-shear Bond Strengths : all experimental groups

decreased as follows: $SM \geq SE \geq SB \geq PL \geq GI$. However, there were no statistically differences between SM and SE, SE and SB, PL and GI (Fig. 5).

In the cervical $\frac{1}{3}$, there were no statistically differences among SM, SE, SB. And PL, GI showed the lower bond strengths (Fig. 6). In the root dentin, the bond strengths of SM were higher than those of all other adhesives and there were no statistically differences among SB, SE, PL and SE, PL, GI. The self-etching adhesive systems, that is, SE and PL, showed the lower bond strengths than conventional adhesive system and self-priming adhesive system with a separate etching step (Fig. 7). As previously described, there were no significant differences of regional

bond strengths for SM, SB, PL and GI. But only for SE, the bond strength to root dentin was significantly lower than those of other areas ($p < 0.05$).

In addition, the bond strengths of coronal dentin (Occlusal $\frac{1}{3}$, Middle $\frac{1}{3}$, Cervical $\frac{1}{3}$) of GI were significantly lower than those of other resin-based adhesive systems. Then, the bond strengths of root dentin were similar to those of SE and PL. That is, there were no statistically differences among SE, PL and GI.

IV. DISCUSSION

The mechanism of adhesion of the current dentin bonding systems is believed to be mediated through the penetration of adhesive resin into superficially decalcified dentin. This bonding to dentin is result of forming a 'hybrid layer', composed of monomers polymerized in the collagen network of dentin to form a micromechanical interlocking.

Many studies have been done about the relation of regional differences of dentin structure and bond strength. Some studies suggested that the theoretical bond strength of dentin bonding agent at any depth could be evaluated by calculating the area occupied by resin tags, the area of inter-tubular dentin that is infiltrated by the resin and the area of surface adhesion^{6,19)}. Sano et al.^{2,17)} showed that significant variation existed between

the tensile bond strength of bovine mid-coronal and that of cervical root dentin. Also some studies suggested that the remaining dentin thickness can be a influencing factor for the bond strength to dentin as bond strengths decrease when dentin depth increase¹⁵⁾. The remaining dentin thickness is related to tubular density and surface moisture. The number of dentin tubules per unit area is less for radicular dentin, it means that the intertubular dentin is greater in the radicular dentin than the coronal dentin. Gianini et al.³²⁾ reported that regional variations in tubule density and the area occupied by solid dentin might modify bond strengths of both conventional and self-etching adhesive systems. Pashley et al.¹⁹⁾ also assumed that the variable density of dentinal tubules and solid dentin was responsible for differences that had been reported for bond strengths made to superficial or deep dentin. And, Nakajima et al.³⁾ suggested that root dentin showed lower bond strength than coronal occlusal dentin to resin composite. Fogel et al.³⁵⁾ reported that the permeability of radicular dentin is much lower than that of coronal dentin. And, tubules in the periphery of human root dentin have been shown not to extend to the surface in many cases³⁴⁾.

The bond strengths recorded in this study showed that significant difference exists between crown and root dentin only in the self-etching adhesive system, Clearfil SE Bond(SE). The bond strength to root dentin was significantly lower than those of crown dentin. It is similar to the results of the previous studies. Yoshiyama et al.²⁾ reported that the self-etching systems showed high initial bond strengths to sound dentin despite of the presence of a thin hybrid layer. But, compromised bond strengths were shown in sclerotic dentin⁴⁾. Michael et al.¹⁷⁾ showed that the bond strength to coronal dentin was greater than to radicular dentin for Clearfil Liner Bond II (self-etching system). This results are thought to be related to the structural differences between crown and root dentin. In the self-etching systems, the primer is a weak acid, so this may be not enough to remove the smear layer in the root

dentin. And, the amount of intertubular dentin is greater, so the etching efficacy of the primer may be reduced. It is known that self-etching primer generally produces a shallow depth of demineralization than the systems with a separate etching phase²²⁾. In addition, Yoshiyama et al.⁴⁾ suggested that the differences in appearance of hybrid layers between apical root dentin and the other regions (coronal, cervical and middle root dentin) were probably due to the fact that root dentin was more sclerotic than the other regions. In this condition, the tubules seem to be occluded with acid-resistant minerals and the intertubular dentin also seems to be acid-resistant, and the acidic primer of self-etching systems may not solubilize enough minerals. Clearfil SE Bond contains a saturated methacrylated phosphate ester, 10-methacryloxydecyl dihydrogen phosphate (MDP) as the acidic resin monomer. It has a relatively higher pH(2.0) and produces milder effect on the hard tissues⁷⁾. Other systems used in this study showed no significant difference between crown and root dentin.

By the way, the self-etching adhesive system, Clearfil SE Bond(SE) showed the comparable strengths to the conventional three-step system, Scotchbond multi-purpose plus(SM) in the coronal dentin. We can say that this result may support the suggestion of the manufacturers that the risk for incomplete impregnation of the demineralized dentin by the adhesive resin is avoided in the self-etching adhesive system(two-step). Nakabayashi and Saimi¹³⁾ suggested that the self-etching primer was very effective in creating diffusion channels while simultaneously promoting monomer impregnation at the same depth in their study. They also reported that hybridization created by this system was free from defects and was continuous from resin to calcium-rich dentin. However, it is still unclear if these materials can produce strong, durable bonds.

The self-priming adhesive system, Single Bond(SB) showed slightly lower bond strength than those of the conventional adhesive system and the self-etching system in the coronal dentin but there were almost no significant differences

among these three systems. In the root dentin, it showed the higher bond strength than those of the self-etching systems, Clearfil SE Bond (SE) and Prompt L-Pop(PL). For this result, we can consider the reports that the use of self-priming system is recommended than self-etching system in the case of intact, unground enamel, because the self-etching systems generally showed compromised bond strengths to intact, unground enamel⁸⁾. By the way, the primer and adhesive resin combine into one solution in this systems. To obtain the ideal bonding, the adhesive resin has to penetrate sufficiently into the exposed collagen to create a hybrid layer. But generally it had been reported that the self-priming adhesive resin could not fully penetrate to the base of the exposed collagen^{21,22)}. This phenomenon may induce nanoleakage. Some recent studies have suggested that combining the primer and adhesive resin into one step may reduce hybridization effectiveness^{4,20)}. All-in-one adhesive system, Prompt L-Pop(PL) showed the lowest bond strength among the dentin bonding systems used in this study. And, there was no regional difference. The results may mean that all-in-one system cannot yet fulfill all requirements for the production of effective adhesive layer¹⁶⁾. By the way, this material contains methacrylated phosphoric acid esters as the acidic components. Tay et al.^{7,8)} reported that Prompt L-Pop(PL) was more aggressive than Clearfil SE primer and it completely solubilized the smear layer and smear plugs. They also said that this primer was aggressive enough to produce mild dissolution of enamel and it was comparable to the etching effect of phosphoric acid on intact, unground enamel. That is, Prompt L-Pop(PL) has a relatively lower pH(1.0) enough to completely dissolve smear layers. Of course, it does not mean that complete dissolution of smear layer produce higher bond strength. Considering this aspect, we can explain the result that Prompt L-Pop(PL) showed comparable bond strength to crown and root dentin differently from Clearfil SE Bond(SE). As previously described, Clearfil SE Bond (SE) showed the lower bond strength to root dentin compared to coro-

nal dentin.

Resin-modified glass ionomers were developed by combining conventional glass ionomer fillers with resin composite and set by means of an acid-base reaction with polymerization of methacrylate functional groups. The mechanical properties of these materials are superior to conventional glass ionomers but not as strong as composite resins. Various trials have been done to improve the properties of resin-modified glass ionomers. To improve the adhesive property, dentin conditioning with polyacrylic acid was recommended. Polyacrylic acid is a weak etchant and it removes the smear layer but does not remove smear plugs in the dentinal tubule. It may permit the HEMA (2-hydroxyethyl methacrylate) in the resin-modified glass ionomers to penetrate the collagen fiber network in the conditioned dentin and also improves the wetting and adaptation of the materials to dentin³⁹⁾. When the resin-modified glass ionomers are bonded to dentin, the chelation reactions occur between ions around the collagen fibers and the polyacrylic acid molecules diffuse. According to this mechanism, collagen-glass ionomer hybrid materials may be formed. According to the previous reports, 'resin-rich layer' or 'absorption layer' was observed in the interfaces between resin-modified glass ionomers and dentin²⁵⁾.

In this study, Fuji II LC(GI) showed the lower bond strengths than those of other dentin bonding systems. But there were no significant difference between the self-etching adhesive systems including all-in-one system and resin-modified glass ionomers in the root dentin. And, the bond strengths showed no regional differences according to the location of dentin. Pereira et al.³⁹⁾ reported that pulpal pressure had a stronger influence on bond strengths and failure modes of resin-modified glass ionomers than regional differences of substrate. Friedle et al.³³⁾ reported that bonding to superficial dentin was stronger than to deep dentin, just as for most dentin bonding systems. In addition, some previous studies suggested that these materials released at least as much as fluoride as conventional glass

ionomers. It has been shown that resin-modified glass ionomers had an effect on inhibition of dental caries equal to that of conventional glass ionomers^{26,31}.

In this study, bond strengths were measured by means of micro-shear bond test. Recently micro-shear bond test was developed and it was useful to measure bond strengths of extremely small areas²³. In the design of micro-shear bond test, the bonding diameter of the specimen has been as small as 0.7mm²⁴. Compared to micro-tensile bond test, the trimming phase of the specimen after the bonding procedure is not needed, so little stress may produce during the preparation of the specimens. In addition, preparation of the specimens for this method is relatively simple. The results from this study showed the minimal variation of the bond strengths to the same location of dentin. It may indicate the reliability of bond strength. McDonough et al.²⁴ reported that micro-shear bond test could be useful to understand the complex interactions of the dentin-polymer interfaces. As previous studies, shear stress has been believed to be a major stress involved in bonding failure of restorative materials. Until recently, shear bond test was routinely used and it needed the preparation of flat surfaces with diameters ranging between 3 and 10mm. In this testing method, it was difficult to measure the regional bond strengths in the same tooth regarding the size of specimen and there was also an increasing incidence of cohesive failures of dentin during testing. It means that true interfacial bond strength between the dentin and adhesives was not being measured. Cohesive failures in dentin observed after shear bond test might be due to the development of uneven stress distribution within the dentin. Therefore, the conventional shear bond test is now questionable.

As the results of this study, the dentin bonding systems without a separate etching step have to be applied carefully in the cavity including both crown and root dentin. In other words, it had better use the conventional or self-priming adhesive systems in that cavity.

V. CONCLUSION

In the clinical situations, bonding is performed on various sites of dentin and has been affected by intrinsic factors of dentin substrate such as regional structural difference, pulpal pressure, sclerotic change, dental caries and so on.

This study was designed to evaluate regional differences of the micro-shear bond strength to dentin of resin-modified glass ionomer (Fuji II LC; GI) and resin-based adhesives (Scotchbond Multi-Purpose Plus; SM, Single Bond; SB, Clearfil SE Bond; SE, Prompt L-Pop; PL). From the results of this study, it can be concluded as follows :

1. In the occlusal $\frac{1}{3}$ of coronal dentin, the micro-shear bond strengths of SE were higher than other adhesives. There was no significant difference between SM and SB. And PL, GI were followed.
2. In the middle $\frac{1}{3}$ of coronal dentin, the micro-shear bond strengths decreased as follows: SM \geq SE \geq SB \geq PL \geq GI. However, there were no statistically differences between SM and SE, SE and SB, PL and GI.
3. In the cervical $\frac{1}{3}$ of coronal dentin, there were also no statistically differences of micro-shear bond strengths among SM, SE, SB. PL and GI showed the lower bond strengths.
4. In the root dentin, the micro-shear bond strengths of SM were higher than other adhesives, and there were no statistically differences among SE, PL, GI. In addition, SE and PL showed the lower bond strengths than the adhesive systems with a separate etching step (SM, SB).
5. Only for SE, the micro-shear bond strengths of root dentin were significantly lower than coronal dentin ($p < 0.05$).
6. The micro-shear bond strengths of coronal dentin (Occlusal $\frac{1}{3}$, Middle $\frac{1}{3}$, Cervical $\frac{1}{3}$) in the GI were significantly lower than other resin-based adhesive systems. But, there were no statistically differences among SE, PL, GI in the root dentin.

This study suggests that self-etching adhesive systems can produce the lower bond strength to

root dentin than other adhesive systems. Therefore, additional dentin treatments may be needed on applying this systems to the cavity including root dentin. And, the use of self-priming adhesive systems also can be recommended than self-etching adhesive systems in this condition.

Key words : Resin-modified glass ionomer, Resin- based adhesives, Dentin, Micro-shear bond strength

REFERENCES

- Duke E.S. and Lindemuth J. : Variability of clinical dentin substrates. *Am J Dent*, 4 : 241-246, 1991.
- Yoshiyama M., Carvalho R.M., Sano H. and Pashley D.H. : Regional bond strengths of resin to human root dentin. *J Dent*, 24 : 435-442, 1996.
- Nakajima M., Takada T., Tagami J. and Hoseda H. : A study on bonding to dentine in various teeth and sites. *Japan J Conserv Dent*, 34 : 266-274, 1991.
- Yoshiyama M., Matsuo T., Ebisu S. and Pashley D. : Regional bond strengths of self-etching/self-priming adhesive systems. *J Dent*, 26 : 609-616, 1998.
- Yoshiyama M., Carvalho R., Sano H., Horner J., Brewer P.D. and Pashley D.H. : Interfacial morphology and strength of bonds made to superficial versus deep dentin. *Am J Dent*, 8 : 297-302, 1995.
- Pashley D.H. : Dentin bonding : Overview of the substrate with respect to adhesive materials. *J Esthet Dent*, 3 : 46-50, 1991.
- Tay F.R. and Pashley D.L. : Aggressiveness of contemporary self-etching systems. I : Depth of penetration beyond dentin smear layers. *Dent Mater*, 17 : 296-308, 2001.
- Perdiago J., Lopes L., Lambrechts P., Van Meerbeek B. and Vanherle G. : Effects of a self-etching primer on enamel shear bond strengths and SEM morphology. *Am J Dent*, 10 : 141-146, 1997.
- Marshall Jr G.W., Marshall S.J. and Kinney J.H. : The dentin substrate : structure and properties related to bonding. *J Dent*, 25 : 441-458, 1997.
- Swift E.J., Perdiago J. and Heymann H.O. : Bonding to enamel and dentin : A brief history and state of the art. *Quint Int* 26 : 95-110, 1995.
- Kubo S., Finger W.J., Moller M. and Podzun W. : Principles and mechanism of bonding with dentin adhesive materials. *J Esthet Dent*, 3 : 62-69, 1992.
- Sano H., Takatsu T. and Ciucchi B. : Nanoleakage : leakage within the hybrid layer. *Oper Dent*, 20 : 18-25, 1995.
- Nakabayashi N., Kojima K. and Masuhara E. : The promotion of adhesion by the infiltration of monomers into tooth substrates. *J Biomed Mater Res*, 16 : 265-273, 1982.
- Carvalho R.M., Fernandes C.A. and Villanueva R. : Tensile strength of human dentin as a function of tubule orientation and density. *J Adhes Dent*, 3(4) : 309-14, 2001.
- Pereira P.N.R., Okuda M. and Sano H. : Effect of intrinsic wetness and regional difference on dentin bond strength. *Dent Mater*, 15 : 46-53, 1999.
- Bouillaquet S., Gysi P. and Wataha J.C. : Bond strength of composite to dentin using conventional, one-step, and self-etching adhesive systems. *J Dent*, 29 : 55-61, 2001.
- Burrow M.F., Sano H., Nakajima M., Harada N. and Tagami J. : Bond strength to crown and root dentin. *Am J Dent*, 9 : 223-229, 1996.
- Pashley D.H. and Carvalho R.M. : Dentine permeability and dentine adhesion. *J Dent*, 25 : 355-372, 1995.
- Pashley D.H., Ciucchi B., Sano H., Carvalho R.M. and Russel C.M. : Bond strength vs. dentin structure: A modeling approach. *Arch Oral Biol*, 40 : 242-271, 1995.
- Vargas M.A. : Interfacial micromorphology and shear bond strength of single-bottle primer/adhesives. *Dent Mater*, 13 : 316-324, 1997.
- Van Meerbeek B., Yoshida Y. and Snauwaert J. : Hybridization effectiveness of a two-step versus three step smear layer removing adhesive materials examined correlatively by TEM and AFM. *J Dent Res*, 78 : 906-911, 1999.
- Phrukkanon S., Burrow M.F. and Tyas M.J. : The effect of dentine location and tubule orientation on the bond strengths between resin and dentine. *J Dent*, 27 : 265-274, 1999.
- Y. Shimada and P. Senawongse : Bond strength of two adhesive systems to primary and permanent enamel. *Oper Dent*, 27 : 403-409, 2002.
- McDonough W.G., Antonucci K.M. and Shimada Y. : A microshear test to measure bond strengths of dentin-polymer interfaces. *Biomater*, 23(17) : 3603-3608, 2002.
- Sidhu S.K. and Watson T.F. : Interfacial characteristics of resin-modified glass-ionomer cements. *J Dent Res*, 77 : 1749-59, 1998.
- Burgess J.O. : Dental materials for the restoration of root surface caries. *Am J Dent*, 8(6) : 342-51, 1995.
- Oilo G. : Bond strength testing-what does it mean? *Int Dent J*, 43 : 492-498, 1993.
- Paulo E.C. Cardoso and Rebento R. Braga : Evaluation of micro-tensile, shear and tensile tests determining the bond strength of three adhesive systems. *Dent Mater*, 14 : 394-298, 1998.
- Sano H., Shono T. and Sonoda H. : Relationship between surface area for adhesion and tensile bond strength -evaluation of a microtensile bond test. *Dent Mater*, 10 : 236-240, 1994.
- Y. Shono and M. Terashita. : Regional measurement of resin-dentin bond strength. a trial of new version of micro-tensile test method. *Proceedings of the Adhesive dent*, 74-84, 1999.
- Dunne S.M., Goolnik J.S., Millar B.J. and Seddon R.P. : Caries inhibition by a resin-modified and conventional glass ionomer cement, in vitro. *J Dent*, 24 : 91-94, 1996.
- Gianini M., Carvalho R.M. and Martins L.R. : The influence of tubule density and area of solid dentin on bond strength of two adhesive systems to dentin. *J Adhes Dent*, 3(4) : 315-324, 2001.
- Friedl K.H., Powers J.M. and Hiller K.A. : Influence of selected variables on adhesion testing. *Dent Mater*, 8 : 265-269, 1995.
- Furseth R. : The structure of peripheral root dentine in young dentine. *Scand J Dent Res*, 82 : 557-561, 1974.

35. Fogel H.M., Marshall F.J. and Pashley D.H. : Effect of distance from pulp and thickness on hydraulic conductance of human radicular dentin. *J Dent Res*, 67: 1381-1385, 1987.
36. Ferrari M., Cagidiaco C.M. and Mason P.N. : Morphologic aspects of the resin-dentin interdiffusion zone with five different adhesive systems tested in vivo. *J Prosthet Dent*, 71 : 404-408, 1994.
37. Inoue S., Vargas M.A., Abe Y. and Yoshida Y. : Microtensile bond strength of eleven contemporary adhesives to dentin. *J Adhes Dent*, 3(3) : 237-45, 2001.
38. Mount G.J. : Glass-ionomer cements : past, present, and future. *Oper Dent*, 19 : 82-90, 1994.
39. Pereira P.N.R., Yamada T., Tei R. and Tagami J. : Bond strength and interfacial micromorphology of an improved resin-modified glass ionomer cement. *Am J Dent*, 10 : 128-132, 1997.

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