The comparison on micro-tensile bond strengths of variable adhesive systems to Class V cavity

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국문초록

5급 와동에서의 수종 접착 시스템의 결합강도에 관한 비교연구

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이 연구는 발거 소구치에서 5급 와동을 형성하여 접착 시스템 및 와동 위치에 따른 상아질에 대한 접착시스템의 미세인장결합강도의 차이를 비교, 연구하였다.

접착방법은 resin modified glass ionomer(GI), compomer(부식여부에 따라 DE 및 DN군으로 분류), 그리고 상아질 접착제인 Single Bond(SB) 및 Clearfil SE Bond(SE)와 복합레진(Clearfil AP X)을 사용한 5개의 실험군으로 분류하였다. 소구치 협측 치경부에 wedge 형태의 와동을 형성하고 5종의 접착 시스템을 제조자의 지시에 따라 적용, 충전하여 시편을 제작하여 미세인장결합강도를 측정하고, One way ANOVA / Duncan's test로 통계분석하였다. SEM 검사는 미세인장결합강도의 시편제작과 동일한 방법으로 시편을 제작한 후 관찰하였다.

실험 결과, 상아질 접착제 및 복합레진(SB, SE)의 미세인장결합강도가 GI보다 높게 나타났고($p<0.05$), 치은측이 교합측보다 더 낮게 나타났으며, GI, DE, SE에서 유의성 있게 낮게 나타났다($p<0.05$). Compomer에서 conditioning 여부(DN, DE)에 따른 변화는 치은측에서만 유의차 있는 것으로 나타났다. SEM 관찰에서, 교합측의 상아세관은 결합계면과 평행하게 주행하였고, 치은측에서는 결합계면에 수직으로 주행하는 것으로 관찰되었다. (J Kor Acad Cons Dent 29(1):1-12, 2004)

주요어 : 5급 와동, 접착시스템, 와동 위치, 미세인장결합강도, 상아질 접착시스템, 복합레진

Ⅰ. INTRODUCTION

With the recent developments in preventive dentistry and periodontology, senior citizens have more and more retained teeth, so the incidence of root caries and non carious cervical lesions have increased, and the demand for restoration of cervical lesions, root dentin defects such as wedge shaped cervical defects and root caries has increased.

Resin modified glass ionomers were introduced in 1988 by adding resin ingredients to the glass ionomer formulation[1]. The indication area of glass ionomers diverse, as there existed conventional and resin modified glass ionomer formulations for temporary, permanent filling, luting of indirect restoration, crowns & bridges, and brackets for orthodontic treatment, sealing of pits and fis

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sures, and for obturating root canals.

And the recently introduced polyacid modified resins, compomer have become popular for Class V restorations because of their fluoride release and good handling and esthetic properties[2,3]. It was developed as a light curing, one component restorative with fluoride release, that combined the major benefits of both resin composite and glass ionomer chemistry. However, recent studies have revealed that the bond strength of compomer to enamel is not satisfactory, with the result being margin discoloration of the enamel.

Since Buonocore[4] introduced the acid etching technique, although adhesion to phosphoric acid etched enamel is reliable and long lasting, adhesion to dentin has been far more challenging because of the complex mineral and organic phases of dentin. The bonding mechanism of adhesive resins to dentin proposed by Nakabayashi[5] was described as micro mechanical due to the impregnation and polymerization in situ of monomers into exposed collagen of demineralized dentin surfaces, creating a hybrid layer.

Modern dental adhesive systems utilized two different methods to achieve micromechanical retention between resin and dentin. The first method attempted to remove the smear layer completely via acid etching and rinsing, total etching technique. The second approach aimed at preserving the smear layer, self etching/ priming technique[6,7].

Contemporary self etching primers have been developed by replacing the separate acid conditioning step with increased concentration of acidic resin monomers[8]. Self etching primers with different degrees of aggressiveness may either completely dissolve or preserve the smear layer. Despite the presence of a thin hybrid layer of about 0.5~1㎛ in thickness high initial bond strength has been reported for sound dentin[9,13].

Conventional testing methods for adhesion require relatively large surface areas for adhesion, which makes it difficult to evaluate the difference of regional bond strengths. A new bond testing procedure called micro tensile bond strength test has been developed recently that permits the measurement of small cross sectional bonded areas[14]. The procedure allows the testing of irregular surfaces such as Class Ⅰ, Ⅱ and Ⅴ restorations. Since this method can measure the bond strength of a relatively small surfaces, it has been widely used to test different dentin substrates[12,15-17]. In this study, this testing method was used to evaluate the regional bond strength of occlusal and gingival floors of cervical wedge shaped cavities.

The purpose of this study was to compare the micro tensile bond strengths and scanning electron microscopy(SEM) appearances of adhesive bonds made to occlusal vs. gingival surfaces of wedge shaped defects of variable adhesive systems. The null hypotheses were that there was difference in the micro tensile bond strengths of these two regions and between adhesive systems.

Ⅱ. MATERIALS AND METHODS

One resin modified glass ionomer; Fuji Ⅱ LC(GC, Tokyo, Japan), one compomer; Dyract AP(Dentsply, Milford, USA), and two dentin bonding systems and one composite resin; Single Bond(3M, St.Paul, USA), SE Bond(Kuraray, Osaka, Japan), Clearfil AP X(Kuraray, Osaka, Japan), were used. The materials, components, manufacturers used in this study listed in Table 1.

Sixty extracted sound human premolars were used for micro tensile bond testing, and five additional teeth were used for SEM examination, which had been stored in normal saline at 4℃.

Wedge shaped cervical cavities, approximately 5mm wide, 3mm long, 3mm deep, were prepared in the buccal cervical cooling(Fig. 1 A).

The prepared teeth were randomly divided into five experimental groups with twelve teeth each. The identification of the experimental groups by adhesive systems are listed in Table 2.

Table 1. Materials used in this study

Products		Main Components	Manufacturer
Resin modified glass ionomer			
Fuji Ⅱ LC		fluoroaluminum silicate glass, polyacrylic acid, HEMA	GC Co. (Tokyo, Japan)
Dentin Conditioner		20% polyacrylic acid with 3% $AlCl_3$	
Compomer			
Dyract AP		resin, strontium-fluoro silicate glass	Dentsply Caulk (Milford, USA)
Prime & Bond NT	Etchant	36% Phosphoric acid	
	Adhesive	UDMA, PENTA, nanofiller, acetone, trimethacrylate resin	
Non Rinse Conditioner		itaconic acid, maleic acid	
Self-etching system			
Clearfil SE Bond	Primer	MDP, HEMA, water	Kuraray Co. (Osaka, Japan)
	Adhesive	dimethacrylate, microfiller, MDP, HEMA	
Self-priming system			
Single Bond	Etchant	37% Phosphoric acid	3M Co. (St. Paul, MN, USA)
	Adhesive	HEMA, Bis-GMA, ethanol, water Polyalkenoic acid copolymer	
Resin composite			
Clearfil AP-X		Barium glass, silicone dioxide, 3.0§ (0.1~15§), 84.5wt%	Kuraray Co. (Osaka, Japan)

Bis-GMA—Bisphenol-A glycidyl methacrylate
HEMA—Hydroxyethylmethacrylate
MDP—methacryloyloxydecyl dihydrogen phosphate

Table 2. Five experimental groups

Group	Materials	
GI	Fuji Ⅱ LC	Dentin Conditioner
DE	Dyract AP	Prime&Bond NT(with Etching)
DN	Dyract AP	Prime&Bond NT(with NRC)
SE	Clearfil AP-X	Clearfil SE Bond
SB	Clearfil AP-X	Single Bond

1. Specimen preparation

In the resin modified glass ionomer, Fuji Ⅱ LC(GC Co., Tokyo, Japan), the 20% polyacrylic acid dentin conditioner was applied for 10second(sec) and the prepared cavities were rinsed for 10sec and gently blot dried. Fuji Ⅱ LC(GI) was applied in the prepared cavities as the procedure recommended by the manufactures.

Compomer, Dyract AP(Dentsply, Milford, USA), in the 'total etch' protocol(DE), the cavities were conditioned with the 36% phosphoric acid gel for 15sec, rinsed for 10sec and dried briefly to keep the dentinal surfaces visibly moist. Then, the self primed adhesive, P&B NT(Dentsply, Milford, USA) was applied onto the etched surface and evaporated the excess of solvent and wait for 20sec. In the Non Rinse Conditioning protocol (DN), the cavity was first conditioned with the

Non Rinse Conditioner(Dentsply, Milford, USA) for 20sec, and then bonded with P&B NT (Dentsply, Milford, USA), without rinsing. Then, the adhesive layer was light cured for 10sec. After the bonding procedures, each cavity was restored with Dyract AP in one increment(i.e., bulk filled).

The self etching primer system (Clearfil SE Bond, SE; Kuraray Co., Osaka, Japan) was applied on the cavity surface of all specimens according to the manufacturer's instruction. The composite resin bulk filled of Clearfil AP X. Composite resin was light cured (Spectrum 800; Dentsply, Milford, USA) for 40sec.

The self priming system, Single Bond(SB, 3M Dental Products, MN, USA), the cavity was acid etched for 15sec with the 37% phosphoric acid gel, rinsed for 10sec and dried briefly to keep the dentinal surfaces visibly moist. Then, the self primed adhesive was applied two successive coats onto the etched surface and evaporated the excess of solvent with 2~5sec air blast. Then, the adhe sive layer was light cured for 10sec. Composite bulk filled of Clearfil AP X. Resin composite was applied as previously described.

Additional resin composite was then applied onto the buccal surface of the tooth covering the restoration and cured for mounting on the micro tensile testing zig. This procedure is a prerequi site to provide sufficient bulk for micro tensile bond strength testing.

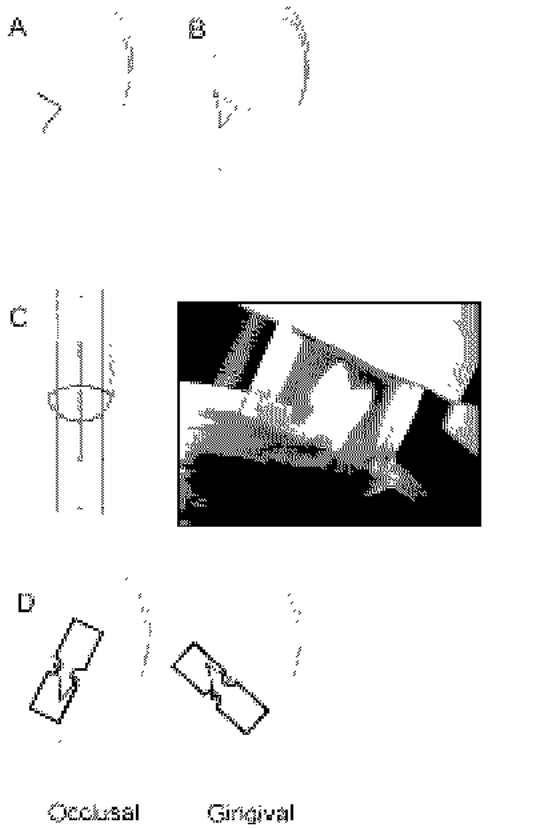

Fig. 1. Specimen preparation for tensile bonding test: A) cavity preparation on buccal cervical area, B) adhesive systems bonding and restoration, additional composite build-up, C) vertical slice with 1mm thickness, D) trimmed specimen with hour glass shape.

2. Micro-tensile bond strength test

All prepared specimens were stored in water at 37℃ for 24hrs and then embedded in the acrylic ring (Diameter 20mm, Height 15mm) with self curing epoxy resin before testing and mounted in a cut off assembly of slow speed diamond saw (ISOMET, Buehler, Lake Bluff, USA) for section ing.

The bonded specimens were then serially sec tioned into two slices approximately 1.0mm thick parallel to the long axis of the tooth using a low speed diamond saw under water cooling(Fig. 1 C).

These sections were then trimmed and shaped to form an hour glass shape with the narrowest portion at the adhesive interface using a superfine diamond point (FG #104R, Shofu, Japan) mount ed in a high speed handpiece under copious water spray(Fig. 1 D). Alternate sections were trimmed to test either the occlusal or gingival walls of each bonded specimen. The adhesive interface trimmed to a cross sectional area, which ranged from 0.95 to 1.05mm^2, was calculated before testing by measuring the diameter and thickness of each specimen. These specimens were then attached to the micro tensile testing zig with a cyanoacrylate adhesive(Zapit, DVA, Lewis Ct. Corona, USA)

which, in turn, was placed in a Testing Machine (EZ Tester, USA) for tensile testing at a crosshead speed of 1mm/min[18].

3. SEM Examination

For the SEM observation of the resin dentin interface, a cervical wedge shaped defect was produced on each tooth in same manner as the micro tensile bond strength test. Each cavity was treated to the bonding procedures mentioned above. The bonded samples were embedded in epoxy resin, then sectioned into two specimens, parallel to the longitudinal axis to the tooth using a low speed diamond saw. Then the cut surfaces were ground with a series of increasingly finer silicon carbide abrasive papers and highly polished with a diamond paste. The specimens were subjected to 10% phosphoric acid treatment for 3 to 5 sec[19 20]. Then specimens were rinsed with water for 15sec and treated with 5% hypochlorite solution for 5 min[21]. After being extensively rinsed with water, the treated specimens were air dried, gold sputter coated, and observed by SEM(S 2300, Hitachi Co., Tokyo, Japan) at 20kvp.

4. Statistical analysis

Overall means and standard deviations (S.D.) of the micro tensile bond strength were calculated for each region: Occlusal wall, Gingival wall

Statistical analysis of the tensile bond strengths was performed using a one way ANOVA and Duncan's test and independent t test at a 95% level of confidence($p<0.05$).

Ⅲ. RESULTS

1. Micro-tensile bond strength

The mean micro tensile bond strengths and standard deviations for each adhesive systems and cavity location are shown in Table 3.

The highest bond strengths measured to both occlusal and gingival wall were obtained with SB group(36.47 MPa and 30.20 MPa), and the lowest bond strengths were obtained with GI group(23.27 MPa and 15.09 MPa).

By comparison of the bond strengths between the adhesive systems, two dentin bonding systems(SB and SE) were higher than resinmodified glass ionomer(GI) on the occlusal wall in bond strengths ($p < 0.05$), and they were higher than resin modified glass ionomer and compomer(GI and DE) on the gingival wall in bond strengths($p < 0.05$). In the all groups, the bond strengths to occlusal wall were higher than those to gingival wall. For GI, DE and SE groups, there were statistically significant differences ($p < 0.05$) when the bond strengths were compared between occlusal and gingival wall. But, for DN and SB groups, there were no statistically significant differences($p > 0.05$).

There was no significant difference to the condi

Table 3. Micro-tensile bond strength of experimental groups (Unit : MPa+SD)

Group	Occlusal wall	Gingival wall
GI	23.27± 8.63^{a}	15.09± 5.10^{a}
DE	29.84±10.54^{ab}	16.52± 4.14^{a}
DN	29.63± 9.94^{abA}	22.53±11.90^{abA}
SE	36.34±11.11^{b}	27.51± 7.99^{b}
SB	36.47±13.39^{bA}	30.20±12.40^{bA}

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

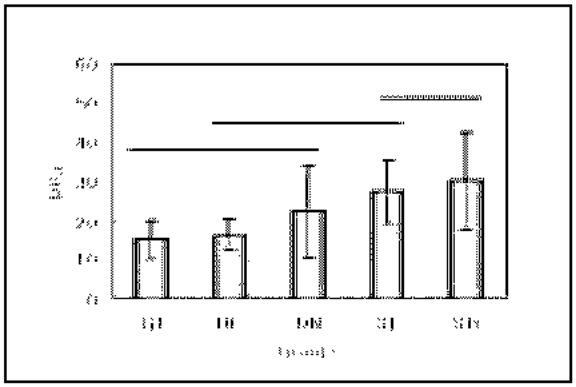

Fig. 2. Micro-tensile Bond Strengths : Occlusal wall

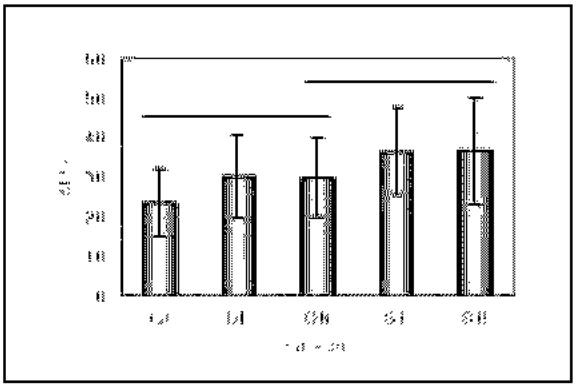

Fig. 3. Micro-tensile Bond Strengths : Gingival wall

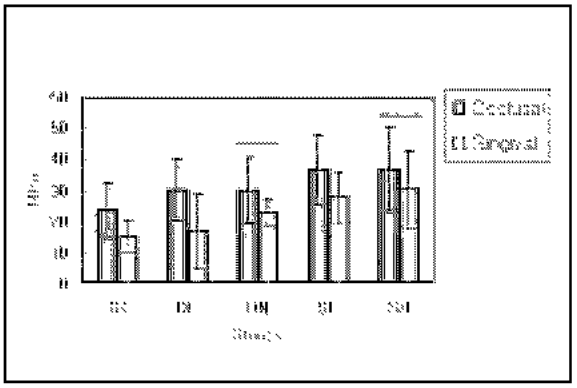

Fig. 4. Micro-tensile Bond Strengths of occlusal and gingival wall

tioning protocol on the occlusal wall between DE and DN($p > 0.05$), in contrast there was signifi cant difference on the gingival wall in bond strengths ($p < 0.05$).

2. SEM Examination

The direction of the dentinal tubules for the occlusal wall was almost parallel to the inter face(Fig. 5~9, A), while for the gingival wall, it was almost perpendicular to the interface(Fig. 5~9, B).

For resin modified glass ionomer, Fuji Ⅱ LC(GI) has adapted well to the conditioned dentin surface. A hybrid like layer was formed between the resin modified glass ionomer and dentin surface, this layer is approximately 2~5㎛ thick(Fig. 5A and 5B).

For the Compomer, Dyract AP, in the etching protocol(DE), formed intertubular and peritubular hybrid layers 2~3㎛ thick(Fig. 6A and 6B). The resin tags exhibited the typical reverse cone shaped appearance indicating that both inter tubular and peritubular dentin were decalcified. Lateral branches were also present. In the Non Rinse Conditioning protocol(DN), a very distinct but thin hybrid layer approximately 1~3㎛ thick was observed(Fig. 7A and 7B). The quality of hybrid layer was poor showing a porous zone along the whole interface of the dentin. The resin tags were thinner and had a less funneled appearance indicating the milder decalcification caused by the etchant. Lateral branches were also formed.

For the SE group, the acidic primer removed most of the smear layer and smear plugs and demineralised the superficial layer of dentine. For occlusal wall, the thickness of the hybrid layer was about 0.5~3㎛, and some areas showed the penetration of resin tags into some tubules(Fig. 8A). For gingival wall, the thickness of the hybrid layer was uniform, at about 4~5㎛, and penetra tion of resin tags into lateral branches of the tubules was also observed, although they were very thin(Fig. 8B).

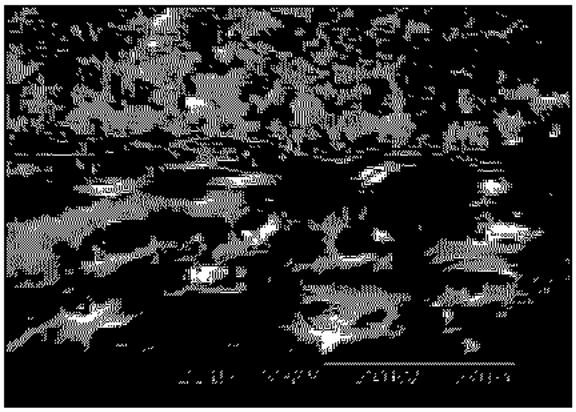

Fig. 5A. SEM photograph of the adhesive interface of the GI group on the occlusal wall.

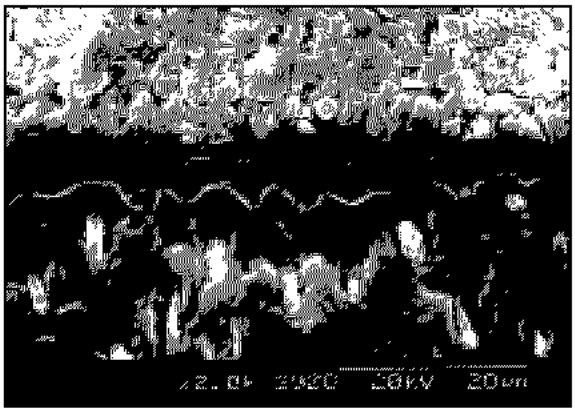

Fig. 5B. SEM photograph of the adhesive interface of the GI group on the gingival wall.

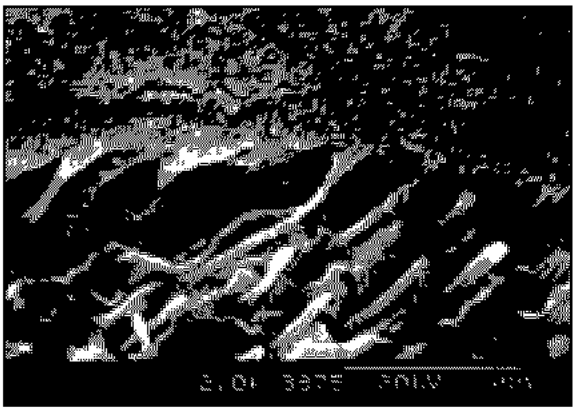

Fig. 6A. SEM photograph of the adhesive interface of the DE group on the occlusal wall.

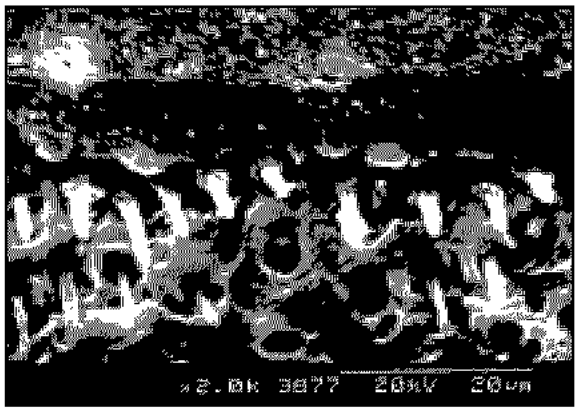

Fig. 6B. SEM photograph of the adhesive interface of the DE group on the gingival wall.

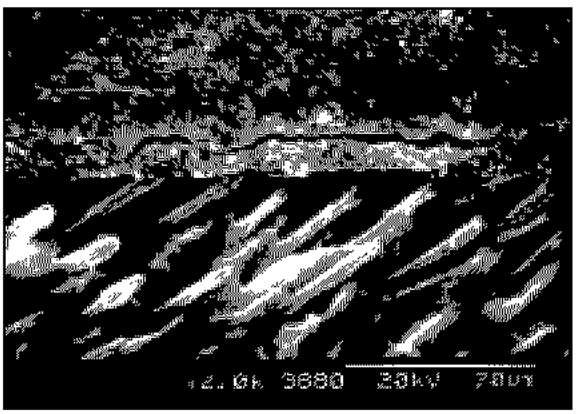

Fig. 7A. SEM photograph of the adhesive interface of the DN group on the occlusal wall.

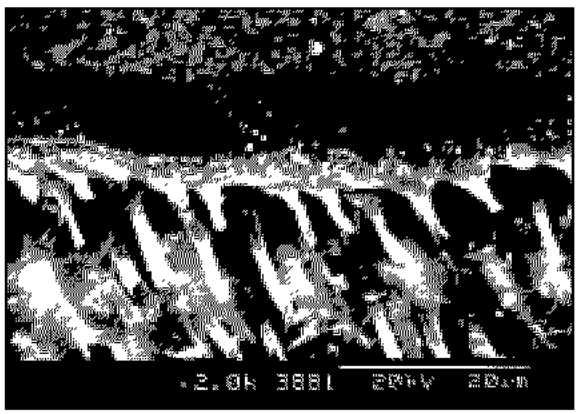

Fig. 7B. SEM photograph of the adhesive interface of the DN group on the gingival wall.

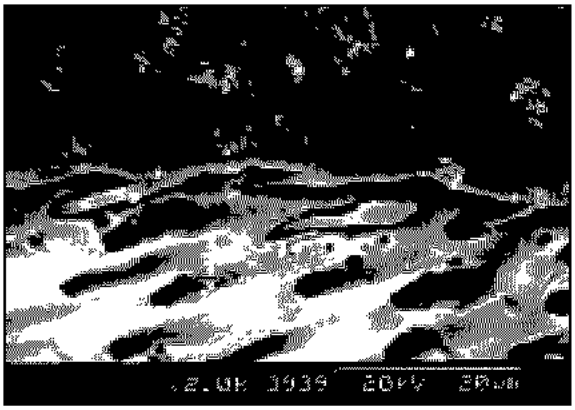

Fig. 8A. SEM photograph of the adhesive interface of the SE group on the occlusal wall.

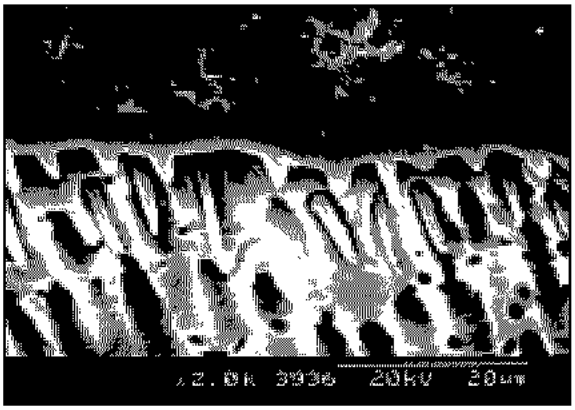

Fig. 8B. SEM photograph of the adhesive interface of the SE group on the gingival wall.

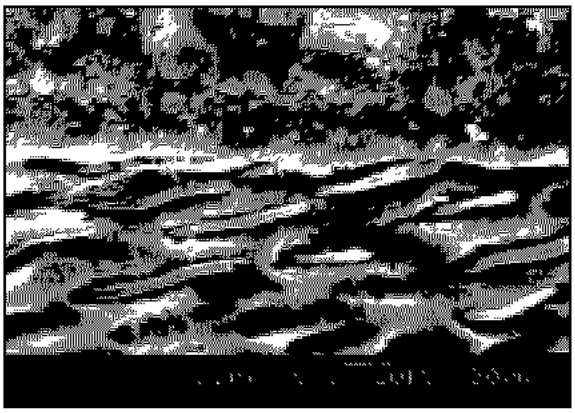

Fig. 9A. SEM photograph of the adhesive interface of the SB group on the occlusal wall.

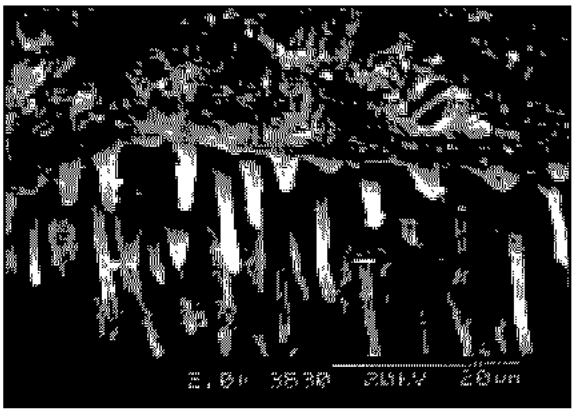

Fig. 9B. SEM photograph of the adhesive interface of the SB group on the gingival wall.

For the SB group to the occlusal wall, the thick ness of the hybrid layer was about 5㎛, and some areas observed showed resin tags in some tubules. The hybrid layer for specimens bonded parallel to tubule axis appeared to be more uni form than that for specimens bonded perpendicu lar to the tubules(Fig. 9A). For the SB group to the gingival wall, a hybrid layer and resin tags could be clealy observed. The thickness of the hybrid layer was about 1~2㎛. The penetration of resin tags into lateral branches of the tubules could be observed(Fig. 9B).

Ⅳ. DISCUSSION

The bond strengths for SB and SE, two dentin bonding systems and composite resin were signifi cantly higher than those reported for GI, resin modified glass ionomer on the occlusal wall. While, the bond strengths for GI and DE were significantly lower than those reported for SE and SB on the gingival wall.

The resin modified glass ionomers were devel oped by combining conventional glass ionomer fillers with resin composite and set by means of an acid base reaction with polymerization of methacrylate functional groups[22]. The mechanical

properties of these materials are superior to conventional glass ionomers but not as strong as composite resins. 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 dentin[23]. When bonded to dentin, the chelation reactions occur between ions around the collagen fibers and the polyacrylic acid molecules diffuse[24]. So, a collagen glass ionomer hybrid materials may be formed. According to previously reported, 'hybrid like layer' was observed in the resin modified glass ionomer and dentin interfaces[25]. In this study, Fuji Ⅱ LC showed the lower bond strengths than those of other adhesive systems. But, some previous studies suggest that these materials release at least as much as fluoride as conventional glass ionomers.

And the recently introduced compomer combines the major benefits of both resin composite and glass ionomer chemistry. However, recent studies have revealed that the bond strength of compomer to the teeth is not satisfactory, with the result being margin discoloration and compomer released significantly less fluoride ions than did the resin modified glass ionomer[26,27]. In this study, compomers(DE and DN) show similar or lower bond strength than two dentin bonding systems with composites.

By the way, the bond strength of DN was significantly higher than those of DE on the gingival wall. It is possible that with Non Rinse Conditioning(NRC) chemical bonding had occurred due to the presence of itaconic acid, which contains carboxylic groups that can adhere to calcium ions of the tooth, thereby contributing to the strength of the bond. Because NRC is not rinsed, the incorporation of the eching debris as fillers might have increased the bond strength[29]. However, there was no significant difference between the bond strengths of DE and DN on the occlusal wall.

In some previous studies, self etching systems showed high initial bond strengths to sound dentin despite of the presence of a thin hybrid layer[13]. It is known that self etching primer generally produces a shallow depth of demineralization than the systems with a separate etching phase[28].

The self priming adhesive system, SB showed slightly higher bond strength than those of the self etching system, SE in the occlusal wall but there were no significant differences among two systems.

In this study, the bond strengths to occlusal wall were significantly higher than those to gingival wall, for GI, DE and SE. There were also statistically significant difference when the bond strengths for each adhesive system were compared between occlusal and gingival wall. But, for DN and SB, there were no statistically significant difference. Phrukkanon et al.[28] compared the micro tensile bond strengths of the Single Bond and an experimental adhesives to dentin as a function of tubule orientation. They suggested that bonding of a self etching primer to sound dentin is independent of the tubular orientation. Yoshiyama et al.[12] measured the regional bond strength of LB(Clearfil Liner Bond) in natural and artificial wedge shaped defects of extracted human teeth. They reported no significant differences between bond strength to the occlusal walls and to the gingival walls. However, Ogata et al.[30] reported that the micro tensile bond strengths of two self etching primer systems were lower at the gingival wall than at the occlusal wall of cervical cavities. They hypothesized that these differences might be explained by the different dentinal tubule orientations.

Since reliable dentin bonding requires optimal hybridization both to intertubular and peritubular dentin, the direction and density of the tubules at the bonding site may affect the quality of the bond[28]. Another factor may be the thickness of

the hybrid layer[31]. With self etching primer systems, the hybrid layer is very thin due to the relatively mild dentin demineralization. In contrast, dentin demineralization by phosphoric acid etching is deeper and the result was that hybrid layer is thicker. However, It has been suggested that the thickness of the hybrid layer is not correlated with bond strength[32-35]. On the other hand, the quality of hybridization is probably the key factor for a bonding between dentin and the resin restorative. Some authors reported that a sound hybrid layer may act as a stress absorbing layer when polymerization contraction stress loads the bonding interface[35-37]. The hybrid layer of compomers(DE and DN) contained more porous areas than two dentin bonding systems with composites, which may affect the strength of the bonded interface.

Many factors can influence the bonding of adhesive systems to dentin. These factors are the dentin substrate, the handling of the material, and the testing methods. Until recently, shear bond tests were routinely used to measure the bonding performance of adhesive systems. Such tests involve the preparation of flat surfaces of dentin with diameters ranging between 3 and 10 mm. However, large human dentin surfaces can be only prepared from crown segments of molars, and molars have great variability in dentin structure and composition. Moreover, the most convenient source of human teeth is unerupted, young third molars, which mainly consist of highly permeable dentin. Bonding procedure on such surfaces will therefore include different substrates resulting in combined bonding patterns. The micro tensile bond strength testing method has recently been developed by Sano et al.[18]. Pashley et al.[38] have stated a number of potential advantages for this methodology: (1) more adhesive failures, fewer cohesive failures; (2) higher interfacial bond strengths can be measured; (3) the ability to measure regional bond strengths; (4) means and variances can be calculated for single teeth; (5) it permits testing of bonds to irregular surfaces; (6) it permits testing of very small areas; and (7) it facilitates examination of the failed bonds by scanning electron microscopy (SEM).

With this study, it became clear that bond strengths of dentin bonding systems and composite resin(SB and SE) to cervical wedge shaped cavity were higher than those reported for resin modified glass ionomer and compomer(GI, DE, and DN) and that the bond strengths to occlusal wall were higher than those to gingival wall.

Ⅴ. CONCLUSION

This study was designed to compare on the micro tensile bond strength (μTBS) of variable adhesive systems to Class Ⅴ cavity, resin modified glass ionomer(GI), compomer(DE and DN), and dentin bonding systems and composite resin(SE and SB). From the results of this study, it can be concluded as follows:

1. The μTBSs for two dentin bonding systems and composite resin(SB and SE) showed higher than resin modified glass ionomer(GI)($p < 0.05$).
2. The bond strengths to the occlusal wall were significantly higher than those to the gingival wall in the GI, DE and SE($p < 0.05$), while, for DN and SB. There were no statistically significant differences between the occlusal and gingival wall($p > 0.05$).
3. There was no significant difference to the conditioning protocol on the occlusal wall between DE and DN, in contrast there was significant difference on the gingival wall ($p < 0.05$).
4. On SEM observation, the direction of the dentinal tubules for the occlusal wall was almost parallel to the interface, while for the gingival wall, it was almost perpendicular to the interface.

In this study the micro tensile bond strength of resin modified glass ionomer is lower than that of composite resin, when caries are thoroughly removed and the cavity is isolated from oral fluid. If the teeth restored with adhesive resin composites, they should be used with their superior

physical properties and excellent bond strengths to tooth tissue.

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