

# BONDING ABILITY OF ADHESIVE RESINS TO CARIES-AFFECTED AND CARIES-INFECTED DENTIN

## CAPACIDADE DE UNIÃO DE RESINAS ADESIVAS À DENTINA AFETADA E INFECTADA POR CÁRIE

Masahiro YOSHIYAMA<sup>1</sup>, Junichi DOI<sup>1</sup>, Yoshihiro NISHITANI<sup>1</sup>, Toshiyuku ITOTA<sup>1</sup>, Franklin R. TAY<sup>2</sup>, Ricardo Marins CARVALHO<sup>3</sup>, David H. PASHLEY<sup>4</sup>

1- Department of Operative Dentistry, Okayama University Graduate School for Medicine and Dentistry, 2-5-1, Shikata-cho, Okayama 700-8525, Japan.

2- Department of Conservative Dentistry, Faculty of Dentistry, The University of Hong Kong, 34 Hospital Road, Hong Kong, China.

3- Department of Operative Dentistry, Endodontics and Dental Materials, Bauru School of Dentistry, University of São Paulo, Al Otavio Pinheiro Brisola 9-75, Bauru, Sao Paulo, 17043-101, Brazil.

4- Department of Oral Biology and Maxillofacial Pathology, School of Dentistry, Medical College of Georgia, Augusta, Georgia 30912-1129, USA.

**Corresponding address:** Dr. Masahiro Yoshiyama, Department of Operative Dentistry, Okayama University Graduate School for Medicine and Dentistry, 2-5-1, Shikata-cho, Okayama 700-8525, Japan. Tel: 81-86-235-6670; Fax: 81-86-235-6674; E-mail: yoshiyam@md.okayama-u.ac.jp

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### ABSTRACT

Hybridized dentin permits dental treatments that were previously impossible with conventional techniques, opening new frontiers in modern adhesive dentistry. We have investigated the adhesive property of current bonding systems to caries-infected dentin by a microtensile bond strength test ( $\mu$ TBS) and transmission electron microscopy (TEM), and suggested that bonding resin could infiltrate into caries-infected dentin partially to embed carious bacteria within hybrid layers. We have named this concept of caries control as modified sealed restoration (MSR). On the other hand, Kuraray Medical Inc. (Tokyo, Japan) has developed an antibacterial adhesive system (ABF, now marketed in USA as Protect Bond). So as to evaluate the effectiveness of ABF on root caries control, we have examine the microtensile bond strengths ( $\mu$ TBS) of ABF to normal versus carious root dentin and the interfacial morphology by a scanning electron microscopy (SEM). ABF could form the hybrid-like structures by infiltrating into the surfaces of the root carious dentin, and the mean value of  $\mu$ TBS of ABF to root carious dentin was 23.0 MPa. These results suggested that MSR combined with ABF might be an advantageous minimal invasive therapy for root caries.

**Uniterms:** Modified sealed restoration; Antibacterial adhesive system; Caries-infected dentin; Microtensile bond strength.

### RESUMO

As técnicas adesivas permitiram inovações nos tratamentos restauradores da atualidade. Este estudo investigou a qualidade de união de diferentes sistemas adesivos ao substrato dentinário afetado e infectado por cárie, através de testes de microtração e microscopia eletrônica de transmissão e varredura (MET). Os resultados sugerem que a resina adesiva pôde infiltrar na dentina infectada e envolver as bactérias na camada híbrida. Esse conceito de controle da cárie foi denominado de "Restauração-Selante Modificada" (RSM). Por outro lado, a empresa Kuraray Med. Inc. (Japão) desenvolveu um sistema adesivo com propriedades anti-bacterianas (ABF), o qual é comercializado nos EUA como Protect Bond. Para avaliar a efetividade do sistema ABF sobre cáries radiculares, empregou-se testes de resistência adesiva e análise por microscopia eletrônica. O sistema ABF foi capaz de formar uma estrutura interfacial semelhante à camada híbrida, através da infiltração na superfície da dentina cariada radicular e os valores de resistência de união foram em média de 23 MPa. Os achados sugerem que a combinação da técnica RSM com o sistema ABF parece ser uma alternativa para o emprego da terapia de mínima invasão em cáries radiculares.

**Unitermos:** Restauração selante modificada; Sistema adesivo anti-bacteriano; Dentina infectada por cárie; Resistência adesiva à microtração.

### INTRODUCTION

Recently, new concepts of treatments for dentin caries by use of adhesive resins and glass-ionomer cements have been proposed. Dramatic changes of the roles of adhesive

dentistry have occurred from the end of the 20<sup>th</sup> century. From the mainly reparative dentistry of the 20<sup>th</sup> century, contemporary dentistry shifts towards a minimal intervention (MI) approach, and contemporary operative treatment incorporates the MI philosophy in cavity design (Peters

and Mclean<sup>13</sup>, 2001). Especially, hybridized dentin permits dental treatments that were previously impossible with conventional techniques, opening new frontiers in modern adhesive dentistry (Nakabayashi and Pashley<sup>11</sup>, 1998).

Traditional treatment of carious teeth involves removal of all carious tooth structure prior to placement of the restorative materials, often sacrificing more structure than necessary. Handleman, et al.<sup>2</sup> (1973) proposed using resin sealants to seal carious pits and fissures following acid-etching. The careful work of Handleman and his colleagues (Jensen and Handleman<sup>8</sup>, 1980) demonstrated that the residual bacteria became dormant and much less viable. This work was followed by a ten year clinical trial by Mertz-Fairhurst, et al.<sup>10</sup>. (1998). They radiographically selected lesions that were beyond the DEJ but no more than half-way to the pulp. The enamel was beveled and no carious dentin was removed. After acid-etching, they placed a chemically-cured radiopaque posterior resin composite. They showed no radiographic progression of the lesions and few viable microorganisms when the resin-sealed lesions were biopsied years after placing resin composites, but further progression of caries in untreated teeth.

Their studies were done with relatively old materials rather than with more hydrophilic contemporary adhesive formulations. If residual bacteria in caries-infected dentin can be embedded by adhesive resins, and if these embedded bacteria become dormant, even caries-infected dentin may be conserved without progression of the caries process. We have proposed this concept as “ Modified Sealed Restoration “ (MSR) as shown in Figure 1. Moreover, if remineralization of caries-infected demineralized dentin occurs after MSR with an antibacterial fluoride-releasing adhesive system (ABF, Protect Bond) developed by Kuraray Medical Inc. (Tokyo, Japan), we may establish the ultra-conservative therapy of caries-infected dentin.

**Resin adhesion to caries-affected and caries-infected dentin**

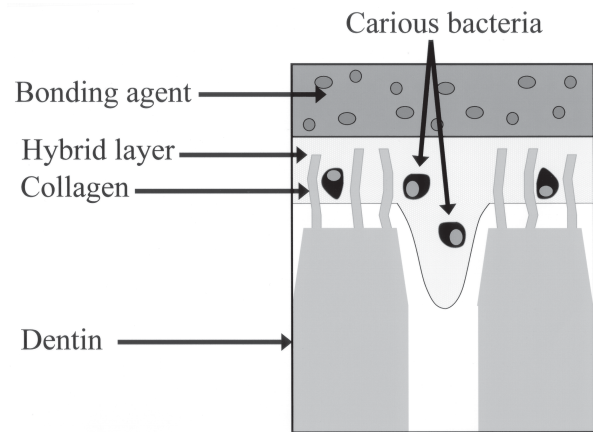
However, so as to establish the ultra-conservative therapy of caries-infected dentin, we should examine the resin adhesion to caries-affected and caries-infected dentin. Recently, a new bond strength testing procedure has been developed which permits the measurement of small (ca. 1mm<sup>2</sup>) cross-sectional bonded areas. It has been called the microtensile bond strength test ( $\mu$ TBS) (Sano, et al.<sup>14</sup>, 1994), and permits the testing of irregular surfaces such as sclerotic and carious dentin. By using  $\mu$ TBS, we were able to evaluate the adhesion of contemporary resin systems to cervical sclerotic dentin (Yoshiyama, et al.<sup>17</sup>, 1996; Tay, et al.<sup>16</sup>, 2000). We have also evaluated the interfacial morphology of two bonding system (Single Bond, 3M and Fluoro Bond, Shofu) to caries-infected dentin, coupled with the measurement of  $\mu$ TBS (Yoshiyama, et al.<sup>22</sup>, 2000), and reported that resin bonds made to caries-affected dentin were lower than to normal dentin using either self-etching primer or conventional adhesive systems. Nakajima, et al.<sup>12</sup> (1999) could demonstrate that very high bond strengths could be

obtained on caries-affected dentin if the dentin is etched with 35% phosphoric acid and the moist bonding technique is employed.

To establish a new dentin caries treatment involving embedding residual bacteria with adhesive resins, we need to evaluate the adhesive properties of bonding resins to caries-infected dentin. Therefore, we have clarified the adhesive property of a self-etching/self-priming system (ABF, Kuraray Medical Inc., Tokyo, Japan)(ABF) to normal, caries-affected and caries-infected dentin using  $\mu$ TBS and transmission electron microscopy (TEM)(Yoshiyama, et al.<sup>20</sup>, 2002). ABF contains 10-methacryloyloxy methacrylate (MDP) as an adhesive monomer. The occlusal surfaces of extracted human third molars with coronal dentin caries were ground perpendicular to the long axis of the tooth to expose a flat surface where the carious lesion was surrounded by normal dentin ( Figure2 ).

The results of the  $\mu$ TBS to normal, caries-affected and caries-infected dentin are shown in Table 1. The  $\mu$ TBS of ABF to normal dentin was about  $44.9 \pm 14.6$  MPa, while the bond strength of ABF to caries-affected dentin was significantly lower ( $25.5 \pm 5.0$  MPa) than that to normal dentin ( $p < 0.05$ ). The  $\mu$ TBS of ABF to caries-infected dentin was only  $15.2 \pm 3.6$  MPa which was significantly lower ( $p < 0.05$ ) than ABF bonds to caries-affected dentin.

TEM observation of the ultrathin sections of the interface from resin-bonded normal dentin showed a thin hybrid layer (less than 1.0  $\mu$ m) that was formed by ABF in the dentin (Figure 3). Higher magnification TEM revealed that smear

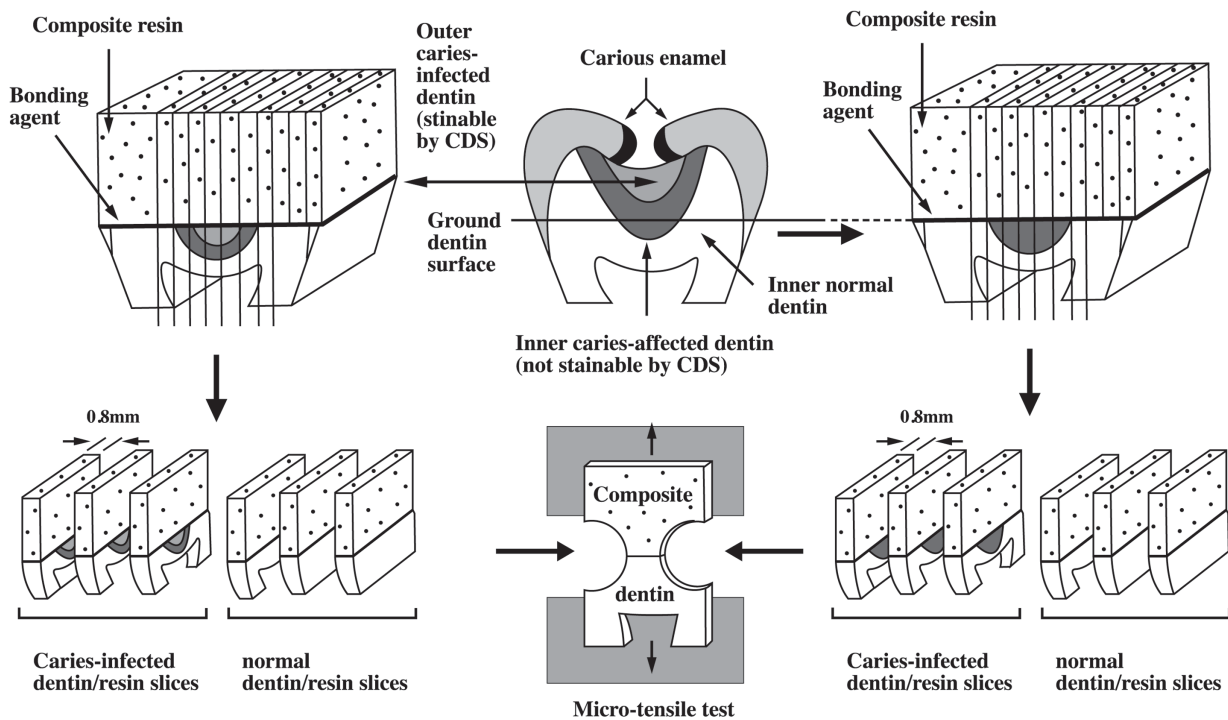


**FIGURE 1-** The concept of Modified Sealed Restoration (MSR). Cariious bacteria may be embedded with hybrid layers.

**TABLE 1-** Tensile bond strength of ABF (Protect Bond) in normal, caries-affected and caries-infected dentin

Normal dentin	44.9 ± 14.6 <sup>a</sup>
Caries-affected dentin	25.5 ± 5.0 <sup>b</sup>
Caries-infected dentin	15.2 ± 3.6 <sup>c</sup>

Values are  $\chi \pm$  S.D. in MPa N=7 in each group. Different superscript lowes ease letters identify statistically significant differences ( $p < 0.05$ ) using Student-Keuls test.

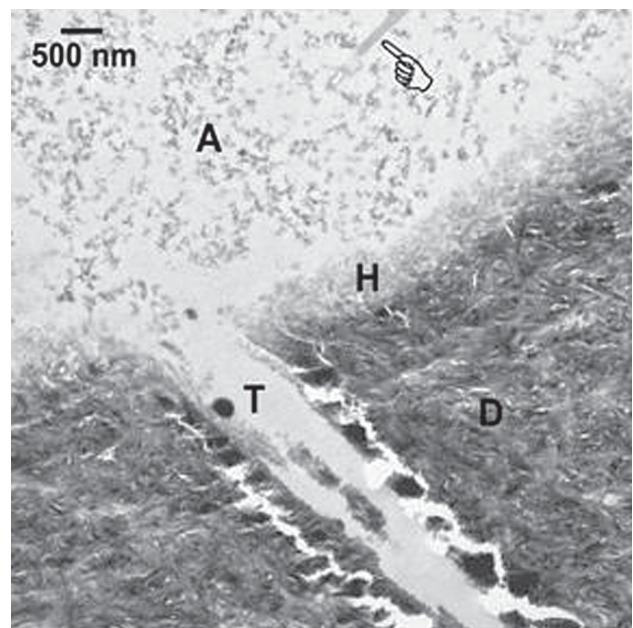


**FIGURE 2-** Schematic showing how carious teeth were prepared for bonding and for micro-tensile test. CDS=caries detective solution ( Kuraray Medical Inc., Japan)

layer was completely dissolved. TEM observation of the interfaces of bonded caries-affected dentin showed a much thicker hybrid layer (6-8  $\mu\text{m}$ ) than was seen in normal dentin. A gradient of resin could also be identified from the surface of the hybrid layer downward, with the base of the hybrid layer poorly identified (Figure 4). Bacteria were rarely observed within the dentinal tubules or on dentin surfaces of the bonded caries-affected dentin. TEM observation of the resin-bonded interfaces of deep caries-infected dentin revealed much substrate variation. In the simplest form, exceptionally thick hybrid layers (ca. 25-30  $\mu\text{m}$ ) could be identified, along with the entrapment of bacteria within some dentinal tubules (Figure 5). Similar to the caries-affected dentin, a gradient of resin could be observed in these thick hybrid layers. Moreover, parts of the dentin surface were not completely wetted by the filled, bonding resin component of the adhesive systems. Other sections revealed thick, erratic hybrid layers that consisted of a superficial layer of completely disorganized and denatured intertubular and peritubular dentin, and an underlying layer of intact hybridized dentin (Figure 6). Bacteria were also trapped within disfigured dentinal tubules.

**Bonding of antibacterial fluoride-releasing adhesive system (ABF) to root carious dentin**

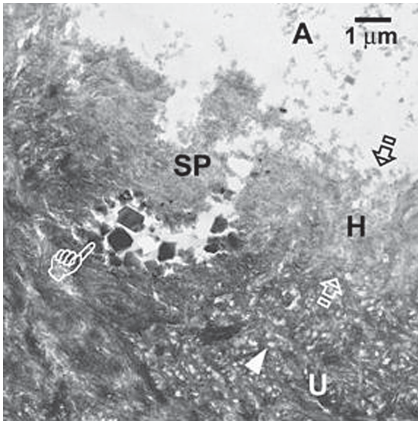
Root caries is an important dental disease, especially because the number of elderly dentate people is increasing (Simons, et al.<sup>15</sup>, 1999). Root surfaces become exposed to oral environment due to gum recession. This gum recession increases with age and the risk of root caries is high because



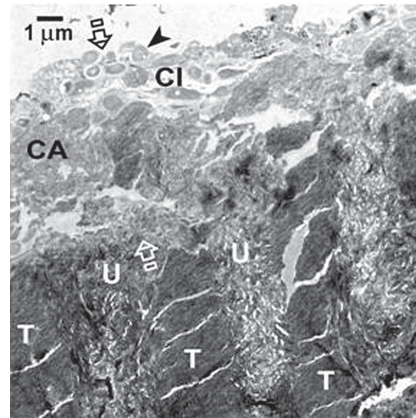
**FIGURE 3-** TEM of the interface of ABF to sound dentin. A thin hybrid layer (H) and a resin tag (T) are seen. A=adhesive rein, D=sound dentin, Pointer=sodium fluoride crystal

of the change in saliva flow rate, the change in cleaning effectiveness and the change in type of bacteria in older patients. Thus, diagnose and rapid treatment of root caries is required for these patients.

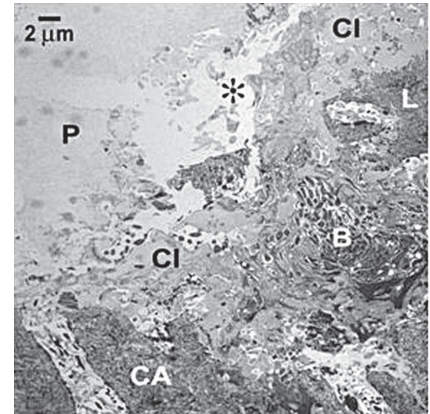
Treatment strategy for root caries include the repair of the decay occurred by the carious progression and the use of antibacterial agents or fluorides for inhibition of the



**FIGURE 4**-TEM of the interface of ABF bonded to caries-affected dentin. Although the hybrid layer (H) was about 3  $\mu\text{m}$  thick, the underlying undemineralized dentin (U) was highly porous (arrow head). The dentinal tubule was covered with a smear plug(SP), and was partially filled with large caries crystals (arrows).



**FIGURE 5**-Bonding of the ABF system to caries-infected dentin showing the variability of the pathological bonding substrate. Stained, undemineralized section. In its simplest form, the hybrid layer(between arrows) consisted of a thin layer of carious-infected dentin (CI) that contained colonized bacteria (arrowhead) that was connected with a layer or caries-affected dentin(U) that was highly porous. The dentinal tubules were completely obliterated with minerals that accounted for the reduction in the permeability in carious dentin.



**FIGURE 6**- Stained, undemineralized section. A thick layer of caries-infected dentin was present, containing loose dentin fragments(L) and bacteria(B). The extent of the hybrid layer could not be clearly discerned. It is likely that the self-etching primer did not reach and infiltrate beyond the layer of caries-infected dentin and did not form a hybrid layer into the underlying caries-affected dentin(CA). A gap(asterisk) was present between the unfilled primer caries-infected dentin. This gap was subsequently filled up with the more electron-lucent epoxy resin that was used for laboratory specimen preparation.

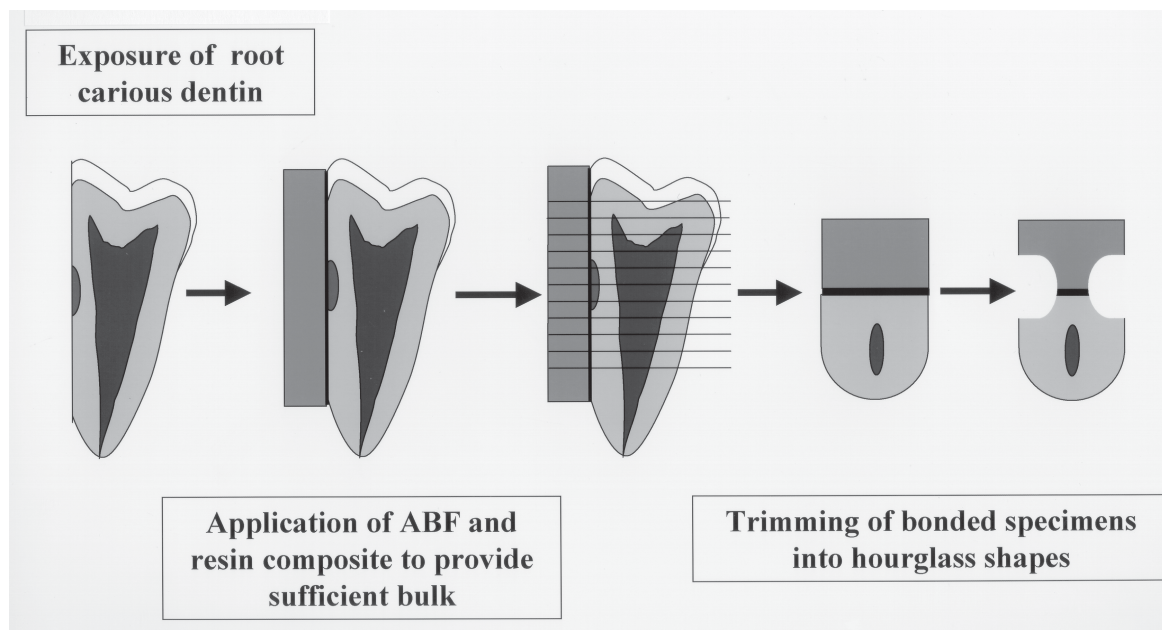
recurrent root caries after the repair. Recently, an antibacterial fluoride-releasing adhesive system (ABF) was developed by Kuraray Medical Inc. (Tokyo, Japan). The self-etching primer in this system contains the antibacterial monomer, methacryloylpyridinium bromide (MDPB) synthesized by combining an antibacterial agent and methacryloyl group, and the adhesive contains sodium fluoride. Imazato, et al.<sup>4,5</sup> (1994, 2001) have reported that MDPB on the surface of a resin-based material showed a bacteriostatic effect and anti-adhesion property against oral streptococci. Thus, the use of this adhesive system for the root carious treatment may inhibit secondary caries occurrence by invasion of bacteria into the resin-dentin interface and adhesion of bacteria on the restorative surface. Fluoride ions released from the adhesive system may also inhibit secondary caries by the remineralization of the dentin around the restoration (Itota, et al.<sup>6</sup>, 2001; Itota, et al.<sup>7</sup>, 2002). In addition, it has been reported that this adhesive system had a good adhesion to normal dentin (Imazato, et al.<sup>5</sup>, 1997).

However, the adhesion of this system to dentin in carious teeth is unknown regardless of the use of this adhesive for the carious treatment. Moreover, even in normal dentin, the bond strength to normal dentin differs in crown and root sites (Yoshiyama, et al.<sup>17</sup>, 1996; Yoshiyama, et al.<sup>18</sup>, 1998).

We have evaluated the  $\mu\text{TBS}$  of ABF to not only normal coronal and root dentin, but also caries-affected and caries-infected root dentin in human carious teeth so as to establish the modified sealed restoration (MSR) combined with ABF to root caries (Doi, et al.<sup>1</sup>, 2004). To expose the normal coronal, normal root, caries-affected root and caries-infected root dentin, the surfaces of human premolars with root

carious lesions were roughly polished using diamond saw (Morita Corp., Tokyo, Japan) with a coolant and then polished with a #600-grit SiC paper under wet condition as shown in Figure 7. The exposed surfaces were distinguished into normal coronal dentin area without dye, normal root dentin area without dye, caries-affected root dentin area with the slightly weak dye or caries-infected root dentin area with the strong dye by caries detector (Kuraray Medical Inc, Tokyo, Japan). The surfaces were then treated with ABF according to manufacturer's instruction, and then covered with resin composite (Clearfil AP-X, Kuraray Medical Inc., Tokyo, Japan) to provide sufficient bulk for  $\mu\text{TBS}$  test. After immersion in 37°C water for 24h, specimens were serially sectioned into multiple slabs about 0.8 mm thick. Each slab was distinguished into normal coronal, normal root, caries-affected root or caries-infected root dentin specimens and then trimmed for  $\mu\text{TBS}$  testing and SEM observation.

Table 2 shows the results of the  $\mu\text{TBS}$  of ABF to the various types of dentin. In normal coronal dentin, ABF could produce quite high bond strength (43.2 MPa), and in normal root dentin, the bond strength of ABF was 33.7 MPa. However, in caries-affected and caries-infected root dentin, the bond strength of ABF were significantly lower than those to normal coronal and root dentin. SEM micrographs revealed that there was the hybrid or hybrid-like layer at the resin-dentin interfaces in all specimens as shown in Figure 8. However, the hybrid-like layers formed in caries-affected root and caries-infected root dentin showed porous



**FIGURE 7-**Schematic diagram showing the methodology used in our study to measure  $\mu$ TBS of ABF to root carious dentin

structures compared to normal coronal and root dentin.

The results of our latest study indicated that the  $\mu$ TBS of ABF to caries-affected root and caries-infected root dentin were significantly lower than those to normal coronal and root dentin. These findings agree with the previous reports that the bond strengths of the current commercial self-etching adhesive systems to caries-affected or caries-infected dentin were significantly lower than that to normal dentin (Yoshiyama, et al.<sup>22</sup>, 2000; Kimochi, et al.<sup>9</sup>, 1999). However, the bond strength of ABF to caries-affected dentin was higher than that of other self-etching adhesive systems reported by several authors (Yoshiyama, et al.<sup>22</sup>, 2000; Nakajima, et al.<sup>12</sup>, 1995). This result suggests that the application of ABF to root caries-affected dentin can contribute to the effectiveness of the restoration. Moreover, ABF adhesive system has an antibacterial effect and fluoride-releasing property. Imazato, et al.<sup>5</sup> (2001) have reported that the incorporation of MDPB into dentin primer could be beneficial for eliminating the residual bacteria in cavities. This report suggests that the use of ABF can eliminate the residual bacteria in caries-affected dentin and caries-infected dentin.

## CONCLUSION

Recent studies have indicated that the bond strengths to caries-affected and caries-infected dentin were lower than that to normal coronal and root dentin even when ABF was used for root carious treatment. However, the antibacterial and fluoride-releasing properties of ABF can be expected to inhibit secondary caries by elimination of the residual bacteria and the invasion of bacteria, and by remineralization of the dentin around the restoration. Further investigations will be necessary for the establishment of the root carious treatment strategy by MSR combined with ABF.

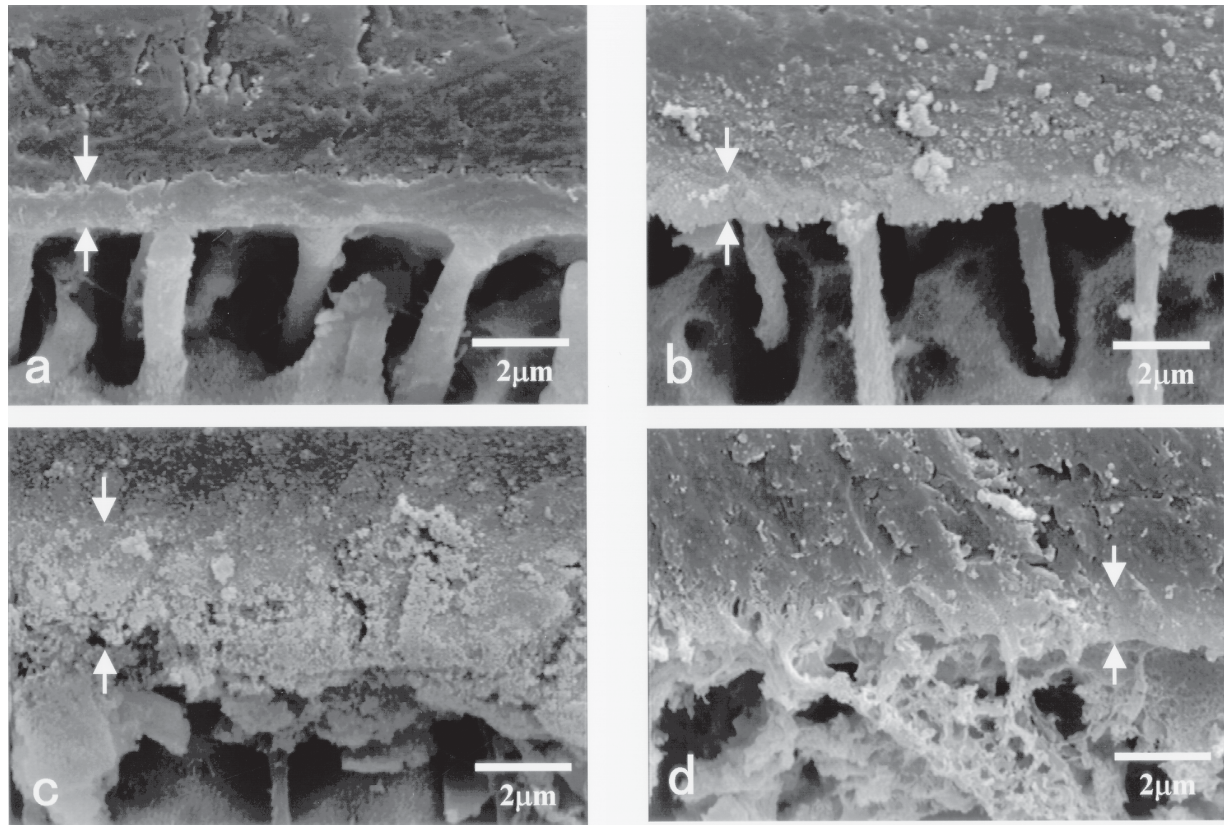
**TABLE 2-** Microtensile bond strength of the ABF to dentin in human root carious teeth

Dentin	Means $\pm$ s.d.
Coronal normal dentin	43.2 $\pm$ 8.1 <sup>a</sup>
Root normal dentin	33.7 $\pm$ 6.7 <sup>b</sup>
Caries-affected root dentin	23.0 $\pm$ 15.3 <sup>c</sup>
Caries-infected root dentin	14.5 $\pm$ 5.5 <sup>d</sup>

Distinct superscript letter indicate significantly different (ANOVA and Fisher's PLSD,  $p < 0.05$ )

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**FIGURE 8** -SEM micrographs of the resin-dentin interfaces in coronal dentin (a),root dentin(b),caries-affected dentin(c) and caries-infected dentin(d). In the coronal dentin, many resin tags and a firm hybrid layer are observed (Figure8a). In the root dentin, a hybrid layer shows the porous structure and the tags structures are thin (Figure8b). In caries-affected and caries-infected dentin, hybrid-layer-like structure shows the porous morphology and tag-like structure is irregular (Figs.8c-d)

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