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# OPERATIVE DENTISTRY

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*Operative Dentistry* publishes articles that advance the practice of operative dentistry. The scope of the journal includes conservation and restoration of teeth; the scientific foundation of operative dental therapy; dental materials; dental education; and the social, political, and economic aspects of dental practice. Review papers, book reviews, letters, and classified ads for faculty positions also are published.

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## GUEST EDITORIAL

### Excellence Isn't a Material

At the recent annual session of the American Academy of Gold Foil Operators, our featured speaker gave a stimulating presentation on the comprehensive restoration of the debilitated dentition. The lecture was particularly well received by the members of the academy, many if not most of whom have been recognized and admired (and sometimes criticized) over the years for their dogmatic dedication to the use of gold in restorative dentistry. What struck me as noteworthy was the fact that the speaker showed very little use of gold in the process of restoration. Virtually everything was full coverage, all ceramic, or metal ceramic. Yet he was warmly received by a group long known for its fastidious adherence to a philosophy of conservation of tooth structure. Paradoxical? Perhaps. Mere superficial professional courtesy? I think not.

The fact that this speaker chose a material different from what many in the audience might have chosen for posterior restorations was a secondary issue. The primary issues were the correct diagnosis, the attention to precise detail in preparation, and the artistic manipulation of the chosen restorative material. What the audience appreciated was the operator's dedication to excellence in his work. Those so dedicated always seem to appreciate others of similar inclination. Of course, there will always be debate in the scientific arena about the attributes and shortcomings of various restorative materials. And so it should remain, as that is what leads to improvement in our materials. But the personal pursuit of excellence is not a debatable issue. It should be an elemental component of the character of every dentist.

More than twenty years ago, Dr Miles Markley explained to me that a casting is rarely as good as it looks, while an amalgam is usually better. His intent was certainly not to cast a disparaging view upon gold, but rather to emphasize that the demands of placing a serviceable casting are far greater than those of an amalgam. Failure to attend to detail at every stage will produce a casting with less-than-ideal fit, causing a progression from leakage to ultimate failure. In the hands of many operators, amalgam is a better choice for the welfare of the patient. I won't dispute that some materials are more

durable than others, but all materials have inherent weaknesses, many of which are purely operator dependent. Once a material is selected (given that it is appropriate for the size and functional requirements of the restoration) the properties of the material take a back seat to the skill and attitude of the operator in determining the ultimate success or failure of the restoration.

So where does that leave us as professionals? Is it truly in our best interest to segregate ourselves into little "Material of the Month" clubs? I don't consider that as counterproductive, as we maintain open minds, recognizing that other groups can contribute meaningfully to the scientific debate. In fact, the small, clinical study club environment has long been hailed as the most effective place to learn excellence in technique. It is only when we carry banners such as "Only gold guys wear white hats," or, "White is right, lead is dead," that we alienate ourselves from our colleagues. We become lemmings, blindly and unalterably following materials, techniques, or schools of thought. We forget that the pursuit of excellence should be our real goal. Excellence is an attitude, not a material. And even though one of our two academies is more material specific than the other, both were, in essence, founded on that premise. We shouldn't forget that.

As I watched the members of the Foil Academy congratulate the speaker that day, I was gratified to see their responses. I knew that they were reacting positively to the general message of diligence and excellence, and not getting unduly sidetracked by one person's espousal of a particular technique or material. That's why I cherish my memberships in both our academies: I get to mix with individuals who are leaders, but who don't muscle their way to the top. They appreciate excellence, regardless of its venue. A significant component of humility is the ability to see the value in the thoughts of others, regardless of their school. I admire that. You fellow members make me proud.

CRAIG BRIDGEMAN

President

American Academy of Gold Foil Operators



## ORIGINAL ARTICLES

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# Thickness and Morphology of Resin-infiltrated Dentin Layer in Young, Old, and Sclerotic Dentin

C PRATI • S CHERSONI • R MONGIORGI  
G MONTANARI • D H PASHLEY

### Clinical Relevance

Sclerotic and old dentin are relatively acid-resistant substrates that resist resin infiltration, especially in superficial dentin. Of the dentin bonding systems tested, Prime&Bond 2.0 produced the thickest resin-infiltrated dentin layers in sclerotic dentin.

### SUMMARY

The purpose of the present study was to evaluate the morphology of the resin tags and the resin-infiltrated dentin layer (RIDL) of several bonding systems in superficial vs deep young, old, and sclerotic human dentin. Dentin was obtained after the removal of occlusal enamel from

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extracted molars. Phosphoric acid gels (35-37%) were used to etch dentin before the application of bonding systems (OptiBond FL; Prime&Bond 2.0; Scotchbond Multi-Purpose Plus and Scotchbond 1; One Step). Each bonded specimen was then sectioned into two halves. One half was polished using a standard procedure to evaluate RIDL thickness and morphology by SEM. The other half was demineralized and deproteinized to evaluate the presence and the morphology of resin tags. RIDL was thinner in superficial dentin than in deeper dentin for all the materials tested regardless of the type of dentin. Sclerotic and old dentin showed thinner RIDL, with short resin tags, and fewer lateral branches than normal dentin.

### INTRODUCTION

Clinical reports suggested that composite resin restorations bonded to sclerotic and old dentin exhibit a high number of clinical failures (Duke & Lindemuth, 1991). Clinical retention performance for many dentin bonding agents (DBAs) in sclerotic and old dentin is lower than in normal young dentin (Heymann & others, 1988). Several in vitro studies (Mixson & others, 1995) suggested that sclerotic dentin differs from normal dentin because of the presence of mineralized precipitates inside the tubules, a high



mineral content (Mixson & others, 1995), and reduced permeability (Tagami & others, 1992). The resin-infiltrated dentin layer (RIDL), as observed by SEM, was described as being thinner in sclerotic than in normal dentin (Inokoshi & others, 1993; Tagami & others, 1993; Van Meerbeek & others, 1994a; Yoshiyama & others, 1996).

The aims of the study were to examine the morphology of RIDL in intertubular dentin and the resin tags and their lateral branches in superficial vs sclerotic dentin using five different dentin bonding agents (DBAs).

Old occlusal dentin was selected from 18 first, second, and third molars of 14 patients (65-78 year-old). The dentin was yellow with no evidence of translucency (North Carolina Dentin Sclerosis Scale of 1) and with no evidence of cervical wear or carious lesions.

Young occlusal dentin was obtained from 30 third molars of young patients between the ages of 22 and 33. The dentin was whitish to very light yellow (North Carolina Dentin Sclerosis Scale of 1).

### Bonding Procedures

The flat prepared dentin specimens were etched with 35 or 36% phosphoric acid gel for 25 seconds, using the acid gel recommended by the manufacturer for that bonding system. The bonded dentin was then restored with five different DBAs, as reported in Table 1. Table 2 reports the number and distribution among the groups of teeth. The manufacturer's directions were followed during the restorative procedures with the exception of the 25-second etching time for the sclerotic dentin groups, instead of the 15 seconds recommended by manufacturers' directions for many DBAs. Each bonded disk was then sectioned in two halves so that each half contained both the superficial and deep levels of bonded dentin.

### Specimen Preparation

One-half of each specimen was stored in 20% phosphoric acid for 70 hours, washed with tap water, and then immersed in 12.5% NaOCl for 24 hours to dissolve all the demineralized dentin matrix. This technique was used to identify the morphology of the resin tags and their associated lateral branches. Each specimen was then gold-coated and inspected under SEM at X1000 to X7500 magnifications. The other

Table 1. Materials Selected for This Study

Material (Bonding System +Composite)	Manufacturers
Prime&Bond 2.0/Dyract	DeTrey/Dentsply, Konstanz, Germany
OptiBond FL/Prodigy	Kerr Corp, Orange, CA 92867
One Step/Z100	Bisco Inc, Itasca, IL 60143
Scotchbond Multi-Purpose Plus/Z100	3M Dental Products, St Paul, MN 55144
Scotchbond 1/Z100	3M Dental Products

## METHODS AND MATERIALS

### Dentin Selection

Occlusal dentin samples were prepared from extracted human teeth using a diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL 60044) under water, then stored in saline solution at 4 °C for 2-3 months.

Superficial dentin was considered to be dentin disks whose central region was within 0.5 mm of the dentinoenamel junction. Deep dentin was considered to be dentin whose flat surface was within 0.5 mm of a pulp horn. Deep dentin was prepared on the same specimens by cutting a step on one-half of the tooth so that each level of dentin was present in each tooth.

Sclerotic occlusal dentin was obtained from 18 extracted molars from 12 old patients (69-75 years old) with cervical wear. The dentin was glassy, yellow or dark-yellow, and the sclerotic region was more than 3.0 mm in diameter. The degree of sclerosis in these teeth was classified as Category 3 or 4 using the North Carolina Dentin Sclerosis Scale as reported by Heymann and Bayne (1993).

Table 2. Sample Distribution: Number of Teeth for Each Group of Materials

	Sclerotic Teeth	Old Teeth	Young Teeth	Total
Prime&Bond 2.0/Dyract	6	6	6	18
OptiBond FL/Prodigy	4	4	6	14
One Step	4	-	6	10
Scotchbond MP Plus/Z100	4	4	6	14
Scotchbond 1/Z100	-	4	6	10

Table 3. Resin-infiltrated Dentin Layer Thickness (RIDL) in Superficial and Deep Dentin, Calculated in Micrometers

Material/Dentin	Prime&Bond 2.0	OptiBond FL	Scotchbond MP Plus	Scotchbond 1	One Step
Sclerotic superficial	0.5-1.8	0.0-1.0	0.2-0.6	-	0.2-0.8
Sclerotic deep	3.0-6.0	0.5-2.0	1.0-2.5	-	0.5-2.0
Old superficial	0.5-2.0	0.5-2.0	0.5-1.5	0.5-1.5	-
Old deep	4.5-7.0	2.5-4.5	3.0-4.0	3.0-5.0	-
Young superficial	2.4-4.0	2.0-3.5	2.0-4.0	3.0-4.0	2.5-3.7
Young deep	4.0-9.0	4.0-6.0	4.0-7.0	5.0-7.7	6.0-8.0

half of the specimens were not demineralized, but the cut surfaces were polished with #600, #1000, #1200, and #4000 abrasive paper, then with diamond paste, treated for 2 minutes with 1.5% NaOCl, washed, ultrasonically cleaned for 2 minutes, and then air dried. Each specimen was sputter-coated with gold and observed with SEM at X2000-5000 magnifications, to permit measurement of RIDL thickness. The morphology of the dentin-bonding system interface was also observed. Six different measurements of RIDL thickness were obtained along the bonded interface in each specimen (from one side to the other side of the disk). These measurements were then averaged for each sample.

RESULTS

Table 3 shows the range of the thickness of RIDL in normal, old, and sclerotic dentin for the three bonding systems tested. The RIDL was consistently thicker in deep than in superficial dentin in all the five groups of materials. Prime&Bond 2.0 had the

thickest RIDL in deep young dentin (ranging from 4.0 to 9.0 microns), which exhibited a granular morphology but had no irregularities or voids (Figure 1) along the resin-dentin interface. Many of the resin tags (especially in Prime&Bond 2.0 samples) observed in the sagittal section showed a “core” of resin surrounded by a larger peripheral RIDL (Figure 1), which indicated that resin monomer had diffused from the tubule lumen into the surrounding demineralized intertubular dentin to form hybridized resin tags.

Sclerotic Dentin

The RIDL was generally thinner in sclerotic dentin than in normal dentin, but it was difficult to measure due to its great variability along the interface (Figures 2-5). In many sclerotic specimens, voids and porosities were observed at the interface between dentin and RIDL or within the RIDL. In some specimens (Figure 2), a gap of about 2-3 microns was observed between RIDL and composite.

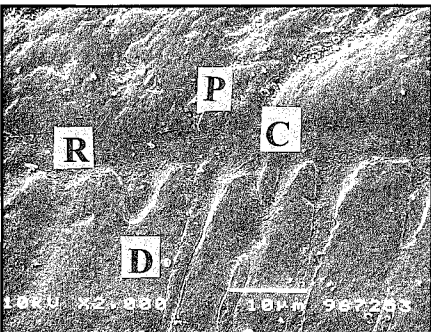


Figure 1. Scanning electron microscope (SEM) image of resin interdiffusion dentin layer (RIDL) formed by Prime&Bond 2.0 (P) in superficial young dentin (D). The RIDL (R) thickness was 8.5 μm. A core of resin is visible inside each tag (C).

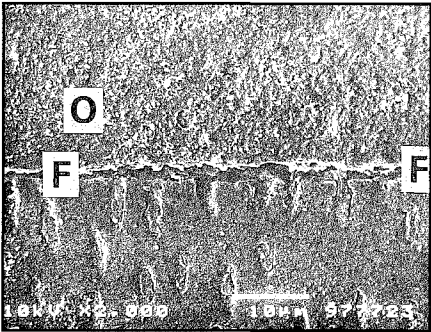


Figure 2. SEM image of RIDL formed by OptiBond FL (O) in superficial sclerotic dentin. RIDL thickness was 0.0-0.0 μm. Note the fracture (F) along composite-RIDL interface.

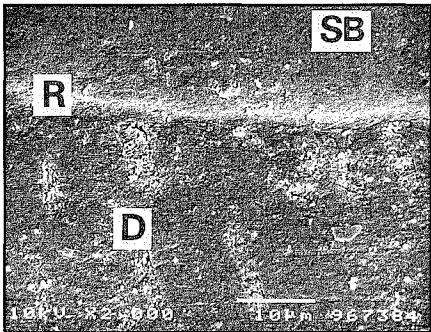


Figure 3. SEM image of sclerotic dentin (D). RIDL (R) formed by Scotchbond Multi-Purpose (SB) is only partially visible. No resin tags are visible, and each dentinal tubule is filled with debris and mineralized casts.

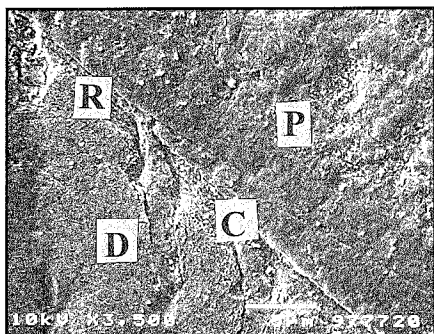


Figure 4. Deep, sclerotic dentin (D). The thickness of RIDL (R) was higher in deep dentin bonded with Prime&Bond 2.0 (P) than in superficial dentin. Note the granular morphology of Prime&Bond 2.0 RIDL and the core of solid resin inside a hybridized resin tag (C).

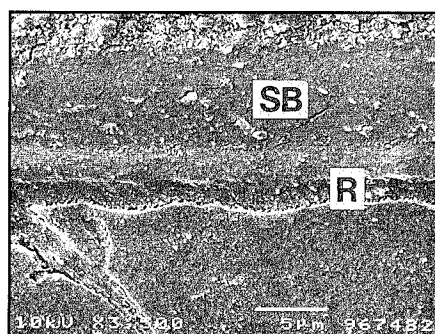


Figure 5. SEM image of Scotchbond Multi-Purpose (SB) sample in sclerotic dentin. RIDL (R) thickness was 2.0-2.5  $\mu\text{m}$ .

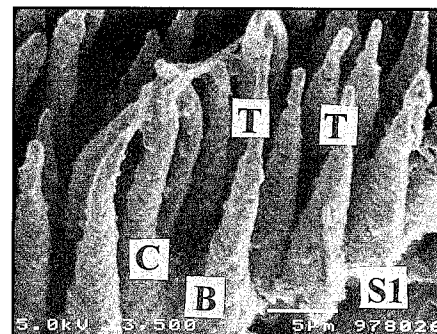


Figure 6. Resin tags (C) formed by Scotchbond 1 (S1) after the removal of all the mineral and collagen tissues in deep old dentin. Many resin tags are visible, with typical tail (T) and large base (B). No resin lateral branches are visible.

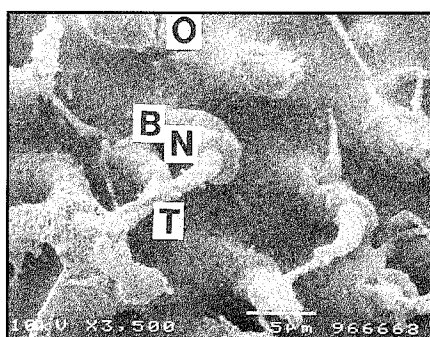
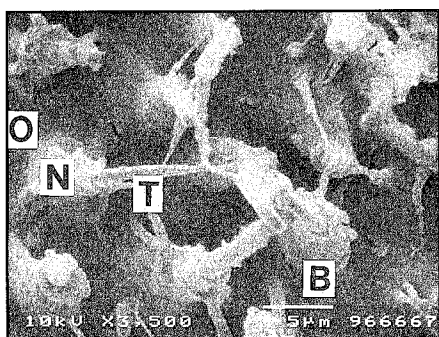
Only a few dentinal tubules in sclerotic dentin were occluded by mineral casts or salt deposits. The patency of the tubules permitted resin to enter the lumen and form resin tags. The resin tag diameters were larger at the top of the dentin and decreased abruptly 3-4 microns deeper in a second step, where the diameter suddenly decreased (Figures 6-9). Resin tags rarely continued much beyond this junction. This type of resin tag configuration was typical for old and sclerotic dentin, while it was rare in normal teeth that exhibited a more gradual taper to the resin tags. Several resin tags of sclerotic dentin were truncated or exhibited short resin tails. Lateral branches to the resin tags were observed only in the initial, larger portion of the resin tags that were located in the original demineralized dentin.

### Old Dentin

In old superficial dentin specimens, the RIDL was thinner than in normal young teeth regardless of the bonding agents. This often was associated with gaps and voids at the resin-dentin interface. In superficial old dentin, the resin tags were shorter than in deep dentin. Resin "tails" were observed in middle to deep dentin.

### Normal Young Dentin

The RIDL was from 2.5 to 7.0  $\mu\text{m}$  thick, depending on the location. Superficial young dentin consistently showed thinner RIDL than deeper dentin (Table 2). The resin tags were longer and more numerous in



Figures 7A & B. Resin tags formed using OptiBond FL (O) after demineralization and deproteinization of dentin. Note the morphology of the tags showing a large base (B), a neck (N), a second enlargement, and an abrupt change in the diameter of the resin tag into a long, thin tail (T).

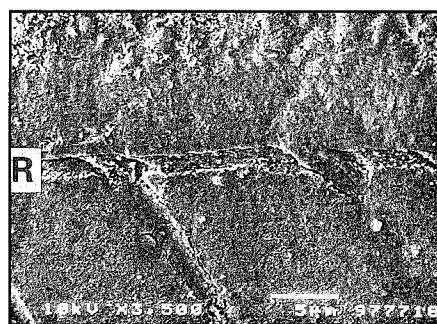


Figure 8. RIDL (R) in old superficial dentin bonded with Prime&Bond 2.0. No tubules or resin tags are visible. Dentinal tubules are filled with mineralized casts.



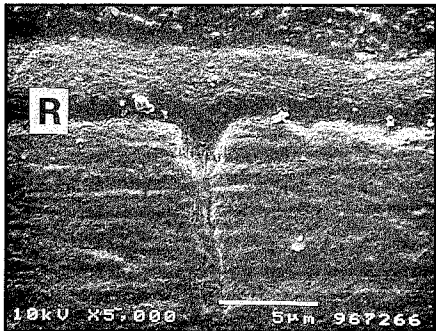


Figure 9. Typical RIDL (R) and resin found in young dentin when bonded with OptiBond FL

young, normal dentin than in old, sclerotic dentin. Lateral branches of resin tags were largely limited to the RIDL. Table 4 reports the number and morphology of lateral resin branches.

DISCUSSION

The RIDL was consistently more than twice as thick in deep, normal dentin compared to superficial, normal dentin (Table 2). We speculate that this is due to the fact that deep dentin is more easily demineralized than superficial dentin because the tubules have a larger diameter (i.e., larger internal surface area) and more or larger lateral branches that permit more rapid radial diffusion of acid from the tubule lumen into the surrounding intertubular dentin. Superficial dentin has fewer, smaller tubules and few lateral branches, limiting demineralization to penetration from the prepared surface (Garberoglio & Brännström, 1976; Pashley, 1991). The present study demonstrated that RIDL was more homogenous, thicker, and more void-free in normal young dentin than in old, sclerotic dentin.

Presumably, this was due to the inability of the acid conditioners used in this study (phosphoric acid gels at 35-37%) to uniformly demineralize sclerotic and old dentin in 25 seconds. Primers and bonding agents can probably infiltrate the entire portion of this shallow demineralized dentin in sclerotic and old dentin. The formation of a thin RIDL may be sufficient to seal dentin, as suggested by many previously published SEM studies (Sano & others, 1994, 1995; Tay & others, 1994), and may be deep enough to form a solid and stable anchorage for a shrinking composite. However, the presence of frequent gaps and voids in sclerotic dentin suggested that this dentin was not an ideal bonding substrate. Such voids were observed most often in superficial sclerotic and old dentin. In some locations of sclerotic and old dentin specimens, no RIDL was observed. We speculate that these areas may be responsible for clinical failures that are often observed in restorations of such dentin. That is, if these gaps exist in vivo, they would serve as stress raisers, causing local concentration of stresses that would propagate cracks through the bonded interface, leading to its failure. The single-bottle bonding system (Prime&Bond 2.0) tested in this study was able to produce thicker RIDLs, suggesting that its permeability in demineralized dentin was high. Because the etching time and the conditioners were similar for all the bonding systems, other mechanisms must be responsible for the thicker/deeper RIDL seen with Prime&Bond 2.0, compared to the other bonding agents. One possible explanation may be that this bonding system is more acidic (its pH is about 1.6) than the pH of the other primers (Scotchbond Multi-Purpose primer is about 3.5). The acidity of Prime&Bond 2.0 may have helped produce a second demineralization of dentin and thus allowed a deeper penetration of primer into the demineralized dentin matrix. This

Table 4. Frequency of Lateral Branches Observed in the Replica Specimens

	Prime&Bond 2.0	OptiBond FL	Scotchbond MP	Scotchbond 1	One Step
Sclerotic superficial	rare	frequent	rare	rare	rare
Sclerotic deep	rare	many	frequent	rare	rare
Old superficial	frequent	frequent	rare	rare	rare
Old deep	frequent	many	frequent	rare	rare
Normal young superficial	many	frequent	frequent	frequent	frequent
Normal young deep	many	many	many	frequent	frequent

Rare = no more than one-two resin lateral branches observed in 10-15% of resin tags; Frequent = more than 50% of resin tags showed two-four resin lateral branches; Many = all the resin tags showed five or more lateral branches.

may have allowed better resin infiltration, especially in sclerotic dentin, into dentinal tubules. Alternatively, it may have been due to its acetone solvent lowering its viscosity, thereby facilitating its penetration into demineralized dentin.

Resin primers and bonding systems were able to penetrate the dentinal tubules of all types of dentin and to fill the lateral branches of the tubules with resin. Resin tag configuration in sclerotic dentin was characterized by a large base (about 3 microns), a small neck 3-4 microns below the surface, a second enlargement, and finally a small-diameter (ca 1  $\mu\text{m}$ ) fine "tail" that extended an additional 5-30  $\mu\text{m}$ . The larger-diameter resin tags filled those portions of the tubules where the acidic conditioners had removed all of the peritubular dentin matrix. The loss of the mineralized peritubular dentin uncovered and enlarged lateral branches, permitting resin to fill them. The small-diameter (ca 1  $\mu\text{m}$ ) resin tag tails probably represent resin that filled those portions of the tubules that still contained mineralized peritubular dentin matrix. Old and sclerotic dentin showed shorter resin tag tails than young dentin samples. Presumably, this was due to the presence of material inside sclerotic dentinal tubules that restricted further resin penetration.

Numerous resin-filled lateral branches were observed extending from the bases of resin tags and from tail portions only in young dentin. The lateral branches may have contributed to a better and deeper resin penetration of bonding system inside demineralized dentin (Pashley & Carvalho, 1997). There is growing evidence that the base of the RIDL is filled with resin monomer via radial diffusion from the tubules by way of lateral branches (Tay & others, 1996a,b). In sclerotic dentin only a few resin lateral branches were detected. This condition may contribute to reducing the diffusion of bonding resin into dentin.

Our study is in agreement with several previous studies (Yoshiyama & others, 1996; Tagami & others, 1993), but only partially with Van Meerbeek and others (1994a), who showed very thin resin-infiltrated layers in sclerotic dentin. However, they limited their observations to cervical root dentin, while we used superficial and deep coronal dentin. They only evaluated the RIDL using Clearfil Liner Bond System (Kuraray, Osaka, Japan), a multi-step bonding system that conditioned dentin with 10% citric acid containing 20% calcium chloride, an etchant that produced more limited etching than 35% phosphoric acid.

There was a high degree of variability of RIDL thickness in old and sclerotic dentin, which suggested that these forms of dentin are very heterogeneous even within the same specimen. Thus, it may not be possible to ever fully characterize sclerotic dentin if it represents a continuum between normal dentin

and dentin in which the peritubular dentin has thickened to the point of obliterating tubule lumens. The use of hardness testing of the dentin adjacent to the bond provided at least some quantitative information on the quality of the sclerotic dentin, while SEM examination (Nakajima & others, 1995; Yoshiyama & others, 1996) provided important qualitative information on resin-dentin bonding.

Sclerotic and old dentin are more clinically relevant bonding substrates than normal dentin, which is usually used in resin bonding studies. In the present study, we extended the etching time from the recommended 15 seconds to 20-25 seconds in an attempt to obtain more demineralization of sclerotic dentin. If the etching time was increased to 35-40 seconds in an attempt to demineralize acid-resistant sclerotic dentin, then one inadvertently over-etches adjacent normal dentin, making it more difficult to prevent its collapse and to completely infiltrate resin monomer to the depth of the demineralization. This increased etching time was apparently insufficient to demineralize superficial or deep sclerotic dentin as well as it did old or young normal dentin. However, even though the RIDLs were thin, they may provide high enough bond strengths to provide retention of class 5 restorations (Van Meerbeek & others, 1994b). Yoshiyama and others (1996) found no difference in tensile bond strength between occlusal vs gingival margins of wedge-shaped defects in sclerotic cervical dentin *in vitro*, even though their RIDLs were of different thicknesses. Thus, there may be no correlation between the thickness of RIDL and resin-dentin bond strength (Finger, Inoue & Asmussen, 1994). Only long-term clinical trials can determine whether extending the etching time on sclerotic dentin would lead to enhanced clinical retention.

## CONCLUSIONS

1. The resin-infiltrated dentin layer (RIDL) was thinner in superficial dentin than in deeper dentin for all the materials tested regardless of the type of dentin.
2. Sclerotic and old dentin showed thinner RIDLs, with short resin tags, and fewer lateral branches than normal dentin.
3. Prime&Bond 2 exhibited the thickest RIDL in deep young and sclerotic dentin.
4. Although sclerotic dentin exhibited a generally thinner RIDL, it was difficult to measure due to its great variability along the RIDL interface.
5. Resin tag configuration differed between old and sclerotic dentin compared to normal dentin.

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# Study of the Shear Bond Strength of Five One-Component Adhesives under Simulated Pulpal Pressure

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## Clinical Relevance

As the storage time increased, combined with simulated pulpal pressure, there was a significant decrease in the shear bond strength for all the adhesive systems used.

## SUMMARY

Recently, several adhesive systems have been introduced that combine the primer and bonding resin in a single bottle. The purpose of this study was to evaluate the bonding efficiency of these one-component adhesives under conditions of simulated pulpal pressure and to determine the influence of storage time on the shear bond strength. One hundred caries-free human molars

were embedded with epoxy resin in cylindrical rubber molds. Flat dentin surfaces at a level 1 mm above the pulpal chamber were obtained and used as the region for bonding. The specimens were randomly assigned to five groups ( $n = 20$ ): (1) Syntac Single, (2) Prime & Bond 2.0, (3) One Step, (4) Single Bond, and (5) OptiBond Solo. Each bonding system was combined with the same composite resin (Herculite XRV). After resin polymerization, half of the samples from each group were tested at 1 week and the other half at 4 weeks. During the bonding procedure and storage time a pulpal pressure of 20 cm of serum was applied. Analysis of the data by one-way ANOVA testing showed that the shear bond strengths were significantly different ( $P < 0.001$ ). OptiBond Solo and Single Bond presented the best results. As the storage time increased there was a significant decrease in the shear bond strength for all the adhesive systems used.

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## INTRODUCTION

The use of adhesive systems in restorative dentistry allows the use of more conservative preparations, the reduction of microleakage in the tooth/restoration

Table 1. Adhesives Tested

Materials	Composition	Manufacturer
Syntac Single	Maleic acid, HEMA, MMPAA	Vivadent, Schaan, Liechtenstein
Prime & Bond 2.0	Elastomeric dimethacrylate resins, PENTA, acetone	Dentsply/DeTrey, Konstanz, Germany
One Step	BIS-GMA, HEMA, BPDM, acetone	Bisco Dental Products, Itasca, IL 60143
Single Bond	HEMA, ethanol, BIS-GMA, dimethacrylates	3M Dental Products, St Paul, MN 55144
OptiBond Solo	HEMA, BIS-GMA, MMPAA, PENTA, acetone, ethanol, water	Kerr Corp., Orange, CA 92667

HEMA: 2-hydroxyethylmethacrylate; MMPAA: methacrylate modified polyacrylic acid; PENTA: dipentaerythritol pentaacrylate monophosphate; BIS-GMA: bisphenol-A glycidyl methacrylate; BPDM: bisphenyl dimethacrylate.

interface, the prevention of recurrent caries and marginal discoloration, and the reduction of postoperative sensitivity (Strassler, 1991). Therefore, one of the primary objectives of researchers is to achieve a strong, durable, predictable union between restorative materials and tooth structure.

Following the introduction of the acid-etched enamel technique (Buonocore, 1955), it was thought that the achievement of the ideal dentin bonding agent would come next; however, the physical and histological properties of dentin have complicated this objective.

Nevertheless, much progress has been made over the last 40 years in this area. The latest dentin-adhesive generation shares some characteristics with its predecessors in that they are multi-use light-curing adhesives, using the total-etch technique, and are compatible with some degree of moisture in the dentin substrate. The considerable attention they have received from clinicians is motivated, to a great extent, by their faster and easier bonding procedure; however, the efficiency of these adhesive materials remains to be determined.

The purpose of this study was to evaluate in vitro the shear bond strength developed by five single-bottle adhesive systems (Table 1) under simulated pulpal pressure, and to determine the influence of storage time on their resistance to fracture.

## METHODS AND MATERIALS

One hundred caries-free human molars were used that had been extracted for orthodontic and

periodontal reasons and stored in an aqueous solution of 5% chlorhexidine before handling.

The samples were prepared according to the bonding protocol developed in the Department of Pathology and Dental Therapeutics, Faculty of Dentistry, University of Granada, Spain (De Haro-Gasquet, 1996). This method uses the dentin surface 1 mm above the pulpal chamber (Figure 1A) to reduce the regional histological variations in dentin structure.

The molars were placed with the occlusal surface in contact with the bottom of the cylindrical inclusion molds, to which they were joined with a drop of cyanoacrylate. A mixture of self-polymerizing resin was prepared and poured around the crown of the molar approximately one-third the way up the root. When the resin was fully polymerized, the resulting cylindrical blocks were removed from the molds.

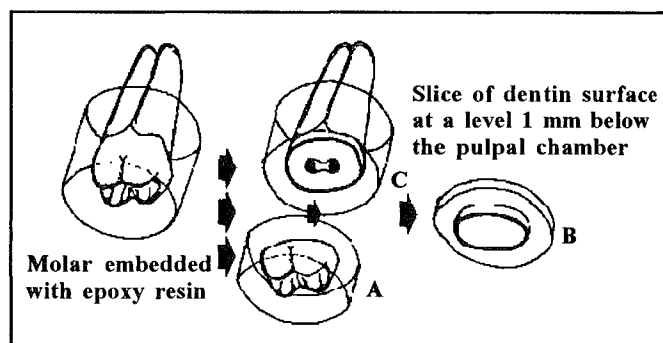


Figure 1A. Schematic diagram of the specimen-obtaining method

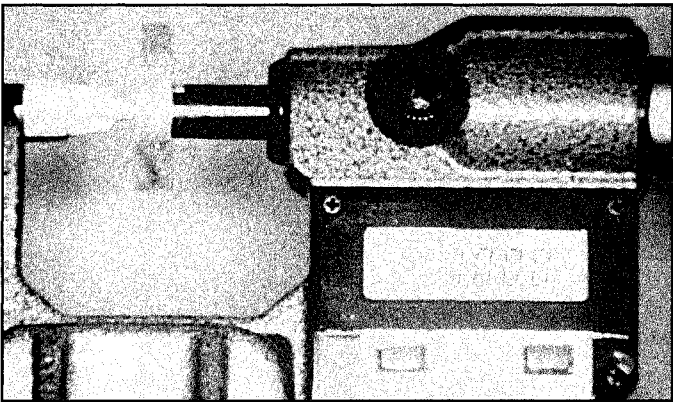


Figure 1B. Measurement of dentin thickness by means of a micrometer

An initial cut was made of the sample, perpendicular to the long axis of the molar, 1 mm from the cementoenamel junction, such that the coronal portion left the pulpal chamber exposed. The pulpal tissue was removed and a 6% solution of citric acid applied for 1 minute.

The distance (D) from the roof of the pulpal chamber to the occlusal surface was then measured with a digital micrometer (Mitutoyo 350 MHN1 – 25 DM, Tokyo, Japan). The distance d ( $d = D - 1 \text{ mm}$ ) was marked, measuring from the occlusal surface in an apical direction. A second cut was made parallel to the first at distance d. When this procedure was completed, the thickness of the remaining dentin was verified as being  $1 \text{ mm} \pm 0.1$  (Figure 1B).

Thus, a flat dentin surface was obtained as the substrate for the bond, in accordance with the “unique free surface technique” previously described by Van Meerbeek and Prati in 1993, which minimizes the effects of the polymerization shrinkage of the composite resin (Finger, 1988).

Furthermore, this procedure attempted to simulate physiological conditions of positive pulpal pressure

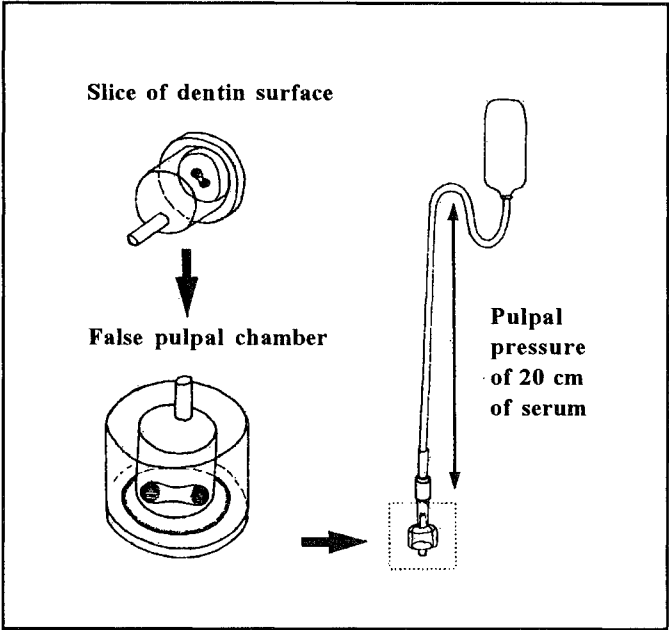


Figure 2. Simulated pulpal pressure system

by creating a false pulpal chamber subsequently filled with a liquid of similar composition to blood serum (Plamaproteinas Grifols, Instituto Grifols, Barcelona, Spain). During the preparation and storage procedures of all the samples, a pulpal pressure of 20 cm of serum was applied, thereby ensuring that the dentin remained under the same conditions of humidity and pressure as occur in clinical use (Figure 2).

The samples were randomly assigned to five groups ( $n = 20$ ). The dentin adhesive system employed in the first group was Syntac Single, in the second group Prime & Bond 2.0, in the third group One Step, in the fourth group Single Bond, and in the final group OptiBond Solo. In all cases the instructions recommended by the manufacturers (Table 2) were followed (Figure 3).

Table 2. Mode of Application				
Material	Etchant	Etch Duration	Number of Layers	Light curing (seconds)
Syntac Single	37% H <sub>3</sub> PO <sub>4</sub> gel	15 minutes	2 (1 + 1)	40 (20 + 20)
Prime & Bond 2.0	37% H <sub>3</sub> PO <sub>4</sub> gel	20 minutes	2 (1 + 1)	20 (10 + 10)
One Step	32% H <sub>3</sub> PO <sub>4</sub> gel	15 minutes	3 (2 + 1)	10
Single Bond	35% H <sub>3</sub> PO <sub>4</sub> gel	15 minutes	2 (2)	10
OptiBond Solo	37.5% H <sub>3</sub> PO <sub>4</sub> gel	15 minutes	1 (brushing)	20



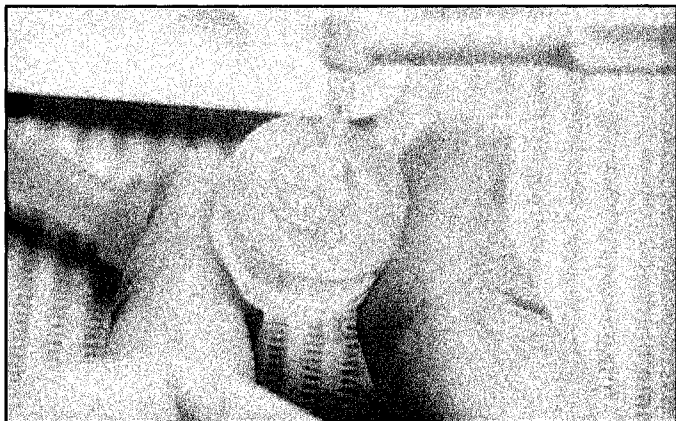


Figure 3. *Application of adhesives on the dentin surface while pulpal pressure is applied*

Finally, a microhybrid resin composite (Herculite XRV, Kerr, Orange, CA 92867) was added onto the conditioned dentin surface, through a cylindrical detachable mold made of vinyl polysiloxane with a diameter of 20 mm, a central hole with a diameter of 5 mm and a depth of 3 mm (Figure 4). The mold was

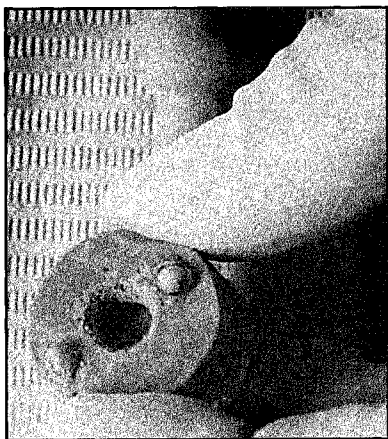


Figure 4. *Cylindrical mold of vinyl polysiloxane*

placed on the dentin surface, and the resin composite was packed into the central cavity two-thirds of the total depth. Then the restorative resin was light cured for 40 seconds using a visible-light-curing unit (Optilux 401, Demetron Research Corp, Danbury, CT 06810) (Figure 5).

Half of the samples from each group were stored in water at a constant temperature of 37 °C for 1 week; the remaining samples were maintained under these

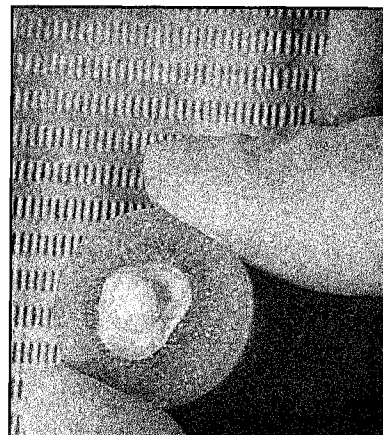


Figure 5. *Microhybrid resin composite added to the dentin surface*

conditions for 4 weeks. The specimens were then submitted to shear bond strength testing performed on an Electrotest machine (Ibertest 500, Barcelona, Spain) (Figure 6). The shear bond strength in MPa was calculated by dividing the shear bond force by the bonded surface area. The debonded specimens were observed under a light microscope to determine the primary mode of fracture.

The data were analyzed using two-way ANOVA (Table 3). Since time x adhesive was statistically significant, separate analyses were made for each time, using one-way ANOVA, and multiple comparisons by the Duncan test. The effect of time within each adhesive was contrasted by using the Mann-Whitney test.

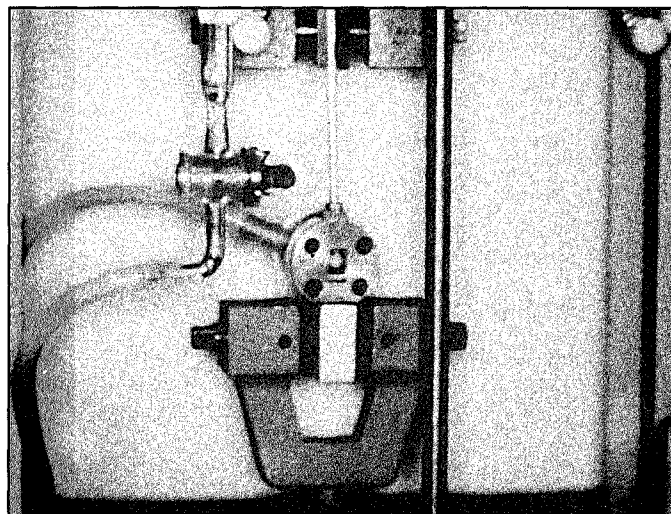


Figure 6. *Electrotest machine*

Table 3. Two-Way ANOVA

Source	Sum of Squares	df	Mean Square	F	P
Adhesive	924.2	4	231.1	21.03	<0.001
Time	729.8	1	729.8	66.42	<0.001
Adhesive x Time	133.8	4	33.5	3.04	<0.05
Residual	988.9	90	10.99		

## RESULTS

The results are displayed in Table 4. The shear bond strengths at 1 week showed that OptiBond Solo and Single Bond were significantly better than other adhesive systems tested. Syntac Single and Prime Bond 2.0 were not statistically different ( $P > 0.05$ ), and One Step was better than Syntac Single. The results after 4 weeks of water storage showed that among OptiBond Solo, Single Bond, and One Step, there were no differences at  $P < 0.05$ . Syntac Single was significantly lower.

Pair-comparisons made by applying the Mann-Whitney U test showed that for all the adhesives tested there was a significant decrease in the shear bond strength related to the storage time of the samples (Figure 7).

The analysis of the failure modes showed that after water storage for 1 week the mode of fracture was cohesive only for the OptiBond Solo and Single Bond systems: either through the composite, cohesive through the dentin, or a mixed failure. In all other groups the mode of fracture was adhesive between the resin and the dentin. After 4 weeks of storage the primary mode of fracture was adhesive for all adhesive systems tested. However, no sample exhibited bond failure in any of the groups during storage or when being mounted in the test apparatus.

## DISCUSSION

Generally, in order to predict the clinical performance of adhesive systems, laboratory tests are performed that evaluate shear bond strength or marginal-seal quality. In both cases, it is difficult to compare the results obtained by different laboratories, since the testing procedures have not been standardized (Retief, 1991; Pashley & others, 1995; Burke & McCaughey, 1995).

The mean resistance to shear for the adhesives

used in our study was below the values (20-30 MPa) presented by the manufacturers or researchers (Pashley & others, 1995). This may be related to the following factors:

(1) As superficial and deep dentin are so differently structured (Paul & Schärer, 1993), the depth of dentin used as the substrate to screen the dentin bonding agents would seem to be very important, and the relative area of dentin occupied by tubules thus decreased from about 45,000/mm<sup>2</sup> at the pulp to about 20,000/mm<sup>2</sup> at the dentinoenamel junction. Moreover, the tubular diameter decreased as it diverged from the pulp: from 1.9  $\mu$ m at the area near the pulp to only 0.8  $\mu$ m for the dentin near the enamel (Garberoglio & Brännström, 1976). This demonstrated that at 1 mm from the pulpal chamber there was a smaller percentage of intertubular dentin available for bonding and an elevated permeability.

(2) As numerous studies (Mitchem, Terkla & Gironas, 1988; Andreus, Koth & Bayne, 1988; Tao & Pashley, 1989; Tagami, Tao & Pashley, 1990; Prati, Pashley & Montanari, 1991; Swift, Perdigao & Heymann, 1995) have established, the pulpal pressure seems to be a determinant in the modification of the adhesive surface, since the dentinal fluid can impede the contact between adhesive and substrate, as well as delay the polymerization of the adhesive resins (Pereira & others, 1997). Despite the manufacturers' instructions that a slight degree of superficial moisture is essential to promote optimal cohesive hybridization, it is possible that the conjunction in our study of high dentin permeability and the application of pulpal pressure may have diluted the primer, thus rendering it less effective (Perdigao & others, 1996). Several studies have shown similar results regarding

Table 4. Results of Shear Bond Strengths

Material	Shear Bond Strength (MPa)		
	1 Week ( $\bar{x} \pm SD$ )	4 Weeks ( $\bar{x} \pm SD$ )	P Value
Syntac Single	5.9 $\pm$ 3.7	2.6 $\pm$ 0.6	<0.050
Prime & Bond 2.0	7.6 $\pm$ 2.6	4.9 $\pm$ 1.8	<0.050
One Step	10.6 $\pm$ 4.6	5.7 $\pm$ 1.7	<0.050
Single Bond	15 $\pm$ 2.9	7.8 $\pm$ 2.6	<0.001
OptiBond Solo	16.3 $\pm$ 4.9	8.0 $\pm$ 4.1	<0.010
$F_{\text{exp}} (4, 45 \text{ gl}) = 13.64, P < 0.001; F_{\text{exp}} (4, 45 \text{ gl}) = 7.91, P < 0.001.$			
$\bar{x}$ = mean; SD = standard deviation; MPa = megapascals.			
Values joined by vertical lines were not significantly different.			

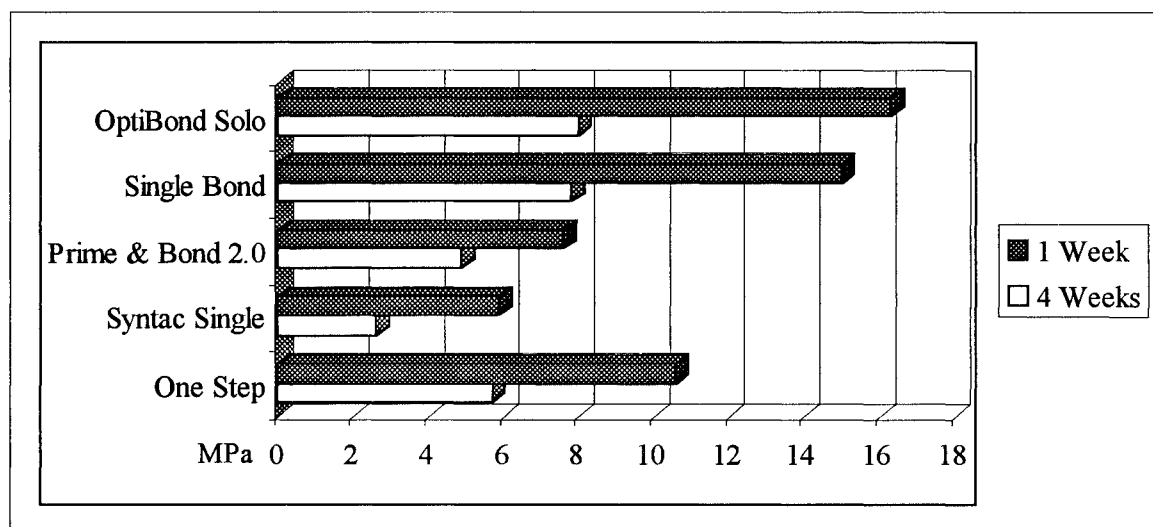


Figure 7. Changes in shear bond strengths

simulated pulpal pressure (Pereira & others, 1997) and dentin histological variations on the shear bond strength by the One Step adhesive (Kanca, 1997).

(3) It is possible that the proteins contained in the liquid used to simulate the dentinal fluid in some way impeded the bonding mechanisms (Nikaido & others, 1995).

In a recent study, Nara and others (1997), comparing the resistance (to traction) of the adhesives Scotchbond Multi-Purpose and Clearfil Liner Bond 2, found no significant differences between the results for samples prepared *in vivo* and *in vitro*. However, the authors did not specify the dentinal depth at which they performed the bond tests; nor did they make clear the time elapsed between bonding the composite and performing the shear bond test. This latter question may be of interest when it comes to considering whether the simulated pulpal pressure was significant, or whether its long-term effects may remain unknown.

For Mason and others (1996), with the hydrophilic dentin bonding systems (Clearfil Liner Bond 2, OptiBond, Scotchbond Multi-Purpose, and All-Bond 2), *in vivo* application did not lead to substantially different results compared to *in vitro* application. We do not believe that our results are comparable—first, because as already stated, the dentinal level chosen as the substrate for the adhesive was a key factor: Mason and others made *in vivo* dentinal cuts at 1.5–2.0 mm from the roof of the chamber, establishing this distance by means of preoperative x-rays. In our opinion, this method may lead to considerable variation in the thickness of the remaining dentin. As explained below, we did not believe that the one-component adhesives used in our study were comparable with their immediate predecessors, since with these

earlier adhesives the separate application of the primer and adhesive may make it possible for deeper, more complete hybridization to occur.

It should be stressed that the purpose of our study was not to ratify how good the bonds were, but simply to compare the behavior of different adhesives under certain experimental conditions. Consequently, negative controls (the same adhesive systems using the same test method with no pulpal pressure at 1 week and at 4 weeks) were not included.

In summary, it is evident that the results of this study cannot be used for directly predicting the clinical performance of the adhesives tested because we did not take into account the three-dimensional nature of cavity preparations; however, by using the same resin composite in all groups, we standardized the effects of polymerization shrinkage, thus enabling comparisons to be made between the different groups.

In absolute terms, the worst results were obtained with Syntac Single, particularly at 4 weeks, where the results were significantly lower than those for the other groups. One Step and Prime & Bond 2.0 performed similarly. These two materials, unlike Syntac, use acetone as a solvent that acts as a water-chaser and helps the diffusion of the primer into the wet dentin substrate (Gwinnett, 1992; Werner, Finger & Fritz, 1996).

Careful analysis of our results revealed that One Step performed relatively better than Prime & Bond 2.0, since the 1-week results for One Step were greater than those for Syntac (whereas there were no significant differences between Syntac and Prime & Bond 2.0), and at 4 weeks the performance of One Step did not differ significantly from that of Single Bond and OptiBond Solo. These relative differences



could be explained by the fact that One Step used more layers than Prime & Bond 2.0, and it has been reported (Nara & others, 1997) that bond strength increases significantly in relation to the thickness of the adhesive layer.

The Single Bond and OptiBond Solo adhesives provided the best results. In both cases, the adhesion promoters and wetting agents are carried in an ethanol solvent.

It is interesting to note that there were significant decreases in the bond strength after 1 month of water storage for all the adhesive systems used. This fact was surprising for us, since presumably these systems ought to provide a more stable bond than systems that have infiltrated the top half of demineralized dentin (Pashley & others, 1995).

SEM evaluation of the resin-dentin interdiffusion zone produced in vivo with other adhesives (Walshaw & McComb, 1996) has shown deficiencies that appear to comprise incomplete surface coverage and incomplete interfibrillar saturation. Recent research has demonstrated the presence of nanometer-sized pores underneath or within the resin/dentin interdiffusion area (Sano & others, 1995). These authors also demonstrated microscopic percolation (nanoleakage) among collagen fibers within the hybrid layer, but its effects on the stability of dentin bonds remain unknown. This, therefore, poses the question as to whether these new one-component adhesives (which combine the primer and the adhesive in a single step) achieve adequate filtration to the full depth of dentin demineralization. Furthermore, the samples were bonded in the presence of serum but stored at 37 °C in water, and therefore, there may have been osmotic pressure expanding the noninfiltrated dentin collagen in the etched but not hybridized dentin layer leading to degradation in bond strength.

Finally, according to our experience, the application of one-component adhesive systems required considerably less time than previous systems. In terms of application and handling, the Single Bond and OptiBond Solo adhesives were the fastest and easiest to use.

## CONCLUSIONS

1. The results obtained for all the adhesive systems tested were below the values that one would expect.
2. The adhesive materials that contain acetone or ethanol as a solvent performed better than the water-based system when used with a moist bonding technique.
3. The Single Bond and OptiBond Solo were the fastest and easiest to use.
4. Further long-term studies are needed to

establish the stability of dentin bonds using recently developed bonding systems.

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# Effect of Dentin Primer Application on Regional Bond Strength to Cervical Wedge-shaped Cavity Walls

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## Clinical Relevance

Multiple primer applications when using Clearfil Liner Bond II improved bond strength to cervical dentin.

## SUMMARY

The purpose of this study was to examine the effect of multiple applications of self-etching primer on regional tensile bond strength ( $\mu$ TBS) to artificial wedge-shaped cavities (i.e., occlusal vs gingival wall). Eighteen extracted noncarious human molars were used to measure  $\mu$ TBS, and four additional teeth were used for scanning electron microscopy (SEM) of the interface.

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Wedge-shaped defects were prepared in the buccal cervical dentin. The teeth were mounted on their distal surface, simulating the supine position of the teeth during dental treatment. The teeth were divided into two groups according to the bonding systems [Clearfil Liner Bond II (LB) or Imperva Fluoro Bond (FB)], and again divided into two subgroups for bonding. One group (Group S) was treated once with the primer according to the manufacturer's instructions. For the other group (Group M), the primer was applied several times for the period recommended by the manufacturer. The adhesive bonding resins were then applied to the cavity walls, and restored with a low-viscosity resin composite (Protect Liner F), and stored in 37 °C water for 24 hours. The resin-bonded teeth were serially sliced parallel to the long axis of the tooth. The adhesive interface of each slice was trimmed alternately at the occlusal or gingival wall, and a microtensile testing method was used to compare resin bond strength to each wall. With both adhesive systems and primer application methods, tensile bond strength to the gingival wall was significantly lower than to the occlusal wall ( $P < 0.05$ ). Tensile bond strength of LB to each cavity wall of a wedge-shaped defect increased

significantly by multiple primer application ( $P < 0.05$ ); however, bond strengths of FB were not significantly different after multiple primer applications.

## INTRODUCTION

Recently, current dentin bonding systems with self-etching primers have been introduced, yielding major improvements in bonding to tooth structure (Chigira & others, 1994; Wang & Nakabayashi, 1991; Watanabe, Nakabayashi & Pashley, 1994; Ikemura, Kouro & Endo, 1996). These adhesive materials attempt to improve the quality of the bond while reducing the bonding procedures. Only one application of etching/primer solution is required in these one-step systems to condition enamel and dentin simultaneously, followed by application of the adhesive resin.

The demand for restoration of root lesions such as wedge-shaped cervical defects and root caries has increased. With the recent use of self-etching primers, these can easily flow from the cavity, leaving a small amount on the walls of a wedge-shaped defect. This is aided by the low viscosity of the self-etching primers. In this case, the dentin surface may not be properly treated by the self-etching primer to produce durable and stable bonds. Ferrari and others (1996, 1997) stated that longer application time of the self-etching primer on dentin created a more intimate interlocking, thus providing an adequate marginal seal. Therefore, it is necessary to devise a suitable method for applying primer to a wedge-shaped defect.

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 the microtensile bond strength test (Sano & others, 1994) has been developed recently that permits the measurement of small (ca 1 mm<sup>2</sup>) cross-sectional bonded areas. The procedure allows the testing of class 1, 2, and 5 restorations. Since this method can measure the bond strength of a relatively small surface, it has been widely used to test different dentin substrates (Nakajima & others, 1995; Yoshiyama & others, 1996; Pereira & others, 1997; Yoshikawa & others, 1997). In this study, this testing method was used to evaluate the regional bond strength within cervical wedge-shaped cavities.

The purpose of this study was to examine the effect of multiple self-etching primer applications of Clearfil Liner Bond II and Imperva Fluoro Bond on regional tensile bond strength to artificial wedge-shaped cavities, as well as to observe the micromorphological appearance of the resin-dentin interface.

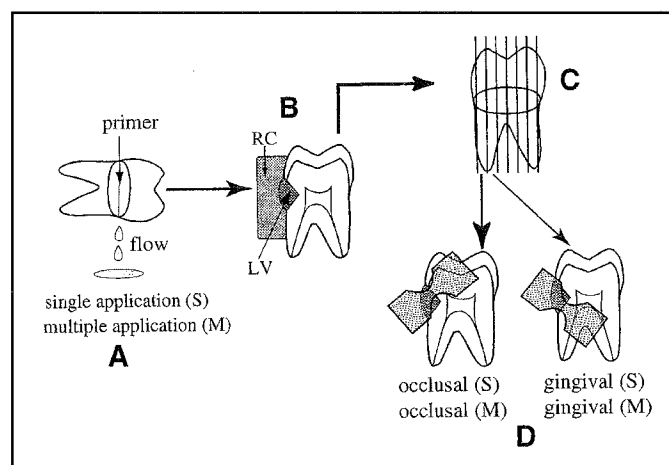


Figure 1. Schematic indication of the methodology used for microtensile bond strength testing. A = Single (S) or multiple (M) primer application to the cavity. The teeth were mounted on their distal surface, so as to permit flow-off of the primer from the cavity. B = the resin bonded teeth; RC = resin composite; LV = low-viscosity resin composite; C, D = sliced specimens that were trimmed to test either occlusal or gingival walls.

## METHODS AND MATERIALS

Eighteen extracted caries-free human upper third molars that were stored frozen were used for microtensile testing (Sano & others, 1994). Wedge-shaped defects were prepared in the buccal cervical dentin by means of a medium-grit diamond point (A-18, GC Ltd, Tokyo, Japan) mounted in a high-speed turbine under copious air-water spray. The dimensions of the cavities were: mesiodistal width—10.0 mm; buccolingival height—5.0 mm; maximum depth—3.0 mm. (Figure 1). The adhesive agents, manufacturers, and batch numbers that were used and the procedures recommended by the manufacturers are listed in Table 1. The identification of the experimental groups and subgroups, methods of primer application, and location of bond strength testing are listed in Table 2. First, the teeth were divided into two groups according to the adhesive systems used for bonding (Clearfil Liner Bond II or Imperva Fluoro Bond). The mesiodistal direction of the cavity was kept parallel to the direction of gravity, simulating the supine position of the teeth during dental treatment, which also permitted the self-etching primer to flow from the cavity (Figure 1). The divided teeth were further subdivided into two groups according to the method of primer application: Group S—primer was put onto the cavity once with a sponge pellet and the teeth were left untouched during the priming time recommended by the manufacturer (LB: 30 seconds, FB: 10 seconds);

Table 1. Adhesive Systems Used for Bonding

System	Ingredients	Code/Lot #	Procedures*	Manufacturer
<b>Clearfil Liner Bond II</b>				
LB-Primer A	Phenyl-P 5-NMSA CQ, ethanol	045	a;b (30 seconds) c;d (20 seconds)	Kuraray Osaka, Japan
LB-Primer B	HEMA water	057		
LB-Bond	MDP HEMA BIS-GMA microfiller	0066		
<b>Imperva Fluoro Bond</b>				
FB-Primer A	water acetone initiator	049604	a;b (10 seconds) c;d (10 seconds)	Shofu Kyoto, Japan
FB-Primer B	4-AET HEMA 4-AETA initiator	049609		
FB-Bond	4-AET HEMA UDMA glass-ionomer filler microfiller	049609		

\*Procedures: a = mix primer; b = apply primer; c = apply adhesive; d = light cure.

Abbreviations: BIS-GMA = bisphenol-glycidyl methacrylate; CQ = camphoroquinone; HEMA = hydroxyethyl methacrylate; MDP = 10-methacryloyloxy methacrylate; NMSA = N-methacryloyl-5-aminosalicylic acid; Phenyl-P = 2-methacryloyloxyethyl-phenyl hydrogen phosphate; UDMA = urethane dimethacrylate; 4-AET = 4-acryloxyethyltrimellitic acid; 4-AETA = 4-acryloxyethyltrimellitate anhydride.

Group M—primer was put onto the cavity several times during the priming time recommended by the manufacturer. Five additional primer applications were made during the recommended priming times of each bonding system. The adhesive bonding resins were then applied to the cavities and light cured. The cavities were filled with a low-viscosity resin composite (Protect Liner F, Kuraray Co, Ltd, Osaka, Japan) (Yoshiyama & others, 1996) and light cured for 40 seconds. After light curing, the specimens were stored in 37 °C water for 24 hours. The enamel, dentin, and resin composite surfaces were then acid etched with 37% phosphoric acid gel (K-etchant, Kuraray), and covered with adhesive resins (Clearfil Photo Bond, Kuraray) to permit adhesion of additional resin composite (Clearfil AP-X, Kuraray) for a microtensile bond test (Figure 1).

The resin-bonded teeth were then serially sectioned into five to six slices approximately 0.7 mm thick parallel to the long axis of the tooth using a low-speed diamond saw (Leitz 1600 Microtome, Leica Instruments, GmbH, Heidelberg, Germany) under water coolant. These sections were then trimmed and shaped to form a gentle curve with the narrowest portion at the adhesive interface using a superfine diamond point (c16ff, GC Ltd, Tokyo, Japan) mounted in a high-speed handpiece under copious water spray. Alternate sections were trimmed to test either the occlusal or gingival walls of each bonded restoration (Figure 1). The bonded surface area, which ranged from 0.95 to 1.05 mm<sup>2</sup>, was calculated before testing by measuring the diameter and thickness of each specimen. These specimens were then attached to the testing device (Bencor-Multi-T,



Table 2. Identification of Groups and Subgroups by Material, Primer Application, Location, and Abbreviations

GROUPS AND SUBGROUPS	ABBREVIATIONS
<b>Group (LB) Clearfil Liner Bond II</b>	
single primer application, occlusal wall	LB-occlusal (S)
single primer application, gingival wall	LB-gingival (S)
multiple primer application, occlusal wall	LB-occlusal (M)
multiple primer application, gingival wall	LB-gingival (M)
<b>Group (FB) Imperva Fluoro Bond</b>	
single primer application, occlusal wall	FB-occlusal (S)
single primer application, gingival wall	FB-gingival (S)
multiple primer application, occlusal wall	FB-occlusal (M)
multiple primer application, gingival wall	FB-gingival (M)

Danville Engineering Co, San Ramon, CA 94583) with a cyanoacrylate adhesive (Zapit, Dental Ventures of America, Corona, CA 91720) which, in turn, was placed in a Universal Testing Machine (Autograph AG-500B, Shimadzu Co, Kyoto, Japan) for tensile testing at a cross-head speed of 1 mm/min (Sano & others, 1994). After fracture of the bonds, all the specimens were visually inspected to determine the mode of fracture. In addition, representative samples were observed using a scanning electron microscope (JXA-840, JEOL, Tokyo, Japan) to confirm the accuracy of the visual inspection.

Statistical analysis of the tensile bond strengths was performed using a two- and three-way ANOVA (LB or FB, occlusal or gingival wall, and single or multiple primer applications) and Fisher's PLSD test at a 95% level of confidence.

For the SEM observation of the resin-dentin interface, four teeth (two for each material) were used, and a cervical wedge-shaped defect was produced on each tooth. Each cavity was treated identically to the bonding procedures mentioned above. The resin-bonded samples were then sectioned into two halves, parallel to the longitudinal axis of the tooth, using a low-speed diamond saw. Each specimen was embedded in epoxy resin (Epon 815, NISSIN EM Co, Ltd, Tokyo, Japan), then the cut surfaces were ground with a series of increasingly finer silicon carbide abrasive papers, and highly polished with a diamond paste (DP-Paste, P, Struers A/S, Copenhagen, Denmark) (6  $\mu$ m, 3  $\mu$ m, 1  $\mu$ m). The samples were subjected to 10% phosphoric acid treatment for 3 to 5 seconds (Gwinnett & Kanca, 1992; Sano & others, 1995). Then specimens were rinsed with water for 15 seconds and treated with 5% hypochlorite solution for 5 minutes (Wang & Nakabayashi, 1991). After being extensively rinsed

with water, the treated specimens were air dried, gold-sputter-coated, and observed by SEM at 10keV. The thickness of the hybrid layers of the resin-dentin interface of each group was measured on each photograph at X4000.

## RESULTS

The resulting microtensile bond strength values ( $\mu$ TBS) and standard deviations are shown in Table 3. Two-way ANOVA analysis revealed that there was a statistically significant correlation between the bonding systems and method of primer application ( $P = 0.002$ ). Three-way ANOVA analysis revealed that there were no statistically significant differences between the bonding systems, the cavity walls, and method of primer application ( $P = 0.8760$ ). With both adhesive systems and methods of primer application, bond strengths to occlusal walls were significantly higher than those to gingival walls ( $P < 0.05$ ). By multiple primer application, bond strength of LB to each cavity wall rose significantly ( $P < 0.05$ ). However, the bond strength of FB indicated no statistical significant difference when altering the method of primer application ( $P > 0.05$ ).

When visually inspected, all specimens showed interfacial adhesive failure. Cohesive failures within dentin or composite were not found. This was confirmed by light microscopic examination (X20). The representative micromorphology of the failure pattern

Table 3. Microtensile Bond Strength Results [mean  $\pm$  1SD (MPa)]

	Occlusal		Gingival
Liner Bond II			
(S)	29.1 $\pm$ 10.8 (n = 15)	$P < 0.05$	17.3 $\pm$ 6.7 (n = 13)
(M)	37.5 $\pm$ 7.9 (n = 12)	$P < 0.05$	26.0 $\pm$ 8.5 (n = 10)
Fluoro Bond			
(S)	31.6 $\pm$ 8.0 A (n = 10)	$P < 0.05$	20.8 $\pm$ 9.6 B (n = 11)
(M)	29.4 $\pm$ 6.9 A (n = 11)	$P < 0.05$	17.8 $\pm$ 6.4 B (n = 10)

n = number of specimens tested.

Groups that are not significantly different are marked with the same alphabetical letter ( $P > 0.05$ ).

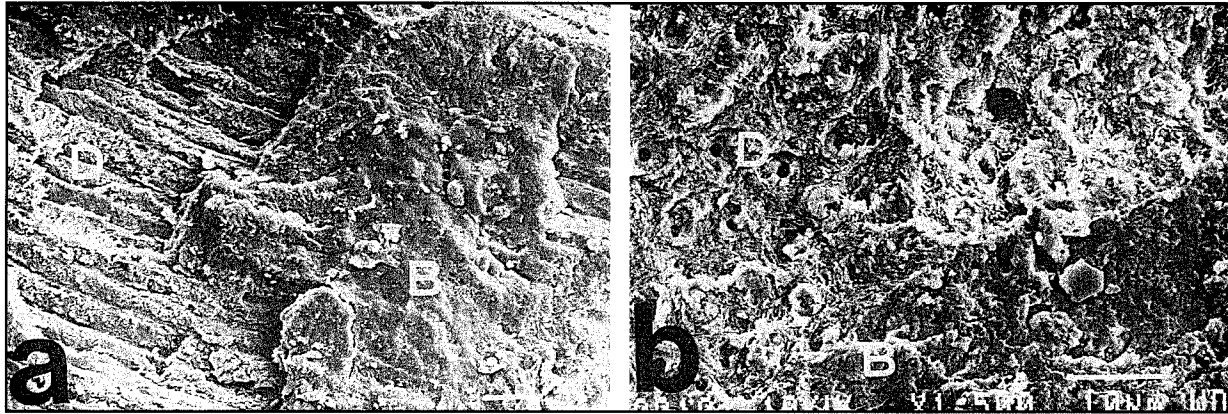


Figure 2. Representative SEM photographs of a fractured specimen. Failure can be seen within bonding resin (B) and dentin (D). A. The direction of the dentinal tubules of the occlusal wall was almost parallel to the interface. B. The direction of the dentinal tubules of the gingival wall was almost perpendicular to the interface. (magnification of A: X472.5, B: X945; bar = 10  $\mu$ m.)

(X750, X1500) was classified as mixed failure within dentin and bonding resin (Figure 2). The direction of the dentinal tubules for the occlusal groups was almost parallel to the interface, while for the gingival groups, it was almost perpendicular to the interface.

The micromorphology of LB-dentin and FB-dentin interfaces are shown in Figures 3 and 4 respectively. For the interfaces treated once with LB-primer [Groups LB-occlusal(S) and LB-gingival(S)], the thickness of the hybrid layer was about 1  $\mu$ m, with narrow and short resin tags that did not fill the tubular orifices completely (Figures 3A and B). For the interfaces treated with LB-primer repeatedly [Groups LB-occlusal(M) and LB-gingival(M)], the thickness of the hybrid layer was about 2  $\mu$ m with thick and long resin tags that exhibited a characteristic funnel cone-shape (Figures 3C and D). For the groups treated with FB, altering the method of primer application made no difference in the interfacial micromorphology. The hybrid layer was not clearly observed (Figure 4).

## DISCUSSION

The influence of multiple primer application on the  $\mu$ TBS was evident for the LB groups, since bond strength to each cavity wall increased significantly (Table 3). The priming time recommended by the manufacturer for LB is 30 seconds to achieve maximum bond strength. Recently, Ferrari and others (1996, 1997) evaluated the effect of priming time of LB on the micromorphology of the resin-infiltrated layer and on marginal sealing ability. They concluded that a longer time of primer application on dentin surface created a more intimate interlocking and provided an adequate marginal seal. In this study, when LB-primer was applied only once to the cavity walls,

the flow-off of the primer from the cavity resulted in an insufficient amount of primer solution on the dentin surface. Because of this, the dentin surface of these groups might not be treated effectively. However, for the groups that received multiple primer application, fresh LB-primer was added. In our study, the shortage of primer on the dentin surface was compensated for by the multiple applications of LB-primer, which contributed to the improvement of adhesive bond strength to dentin. The differences between the SEM appearances of the adhesive interface of LB(S) and LB(M) confirmed this concept (Figure 3). The fact that the resin-infiltrated layer of LB(S) was about 1  $\mu$ m could be explained by the fact the LB-primer was applied only once, which was insufficient to dissolve the smear layer, thereby obstructing the infiltration of the bonding resin into the demineralized dentin. Meanwhile, the hybrid layer of LB(M) showed thick resin tags with a characteristic funnel cone-shape. It is highly likely that the LB-primer dissolved the smear layer completely, and the bonding resin was able to penetrate sufficiently into the demineralized dentin.

On the other hand, multiple primer applications had no influence on the  $\mu$ TBS when the cavities were treated with FB-primer (Table 3). The SEM appearances of the FB groups did not show remarkable differences by the alteration of primer application method. For Group (M) of each bonding system, the frequency of primer addition during the priming time of LB and FB was the same. The priming time of FB-primer (10 seconds) was much shorter than LB-primer (30 seconds). Yoshiyama and others (1998) reported that LB and FB formed thin hybrid layers of about 1.0 mm thick in cervical dentin, and that the resin tags produced by FB were shorter in length but larger in diameter than those produced by LB. They

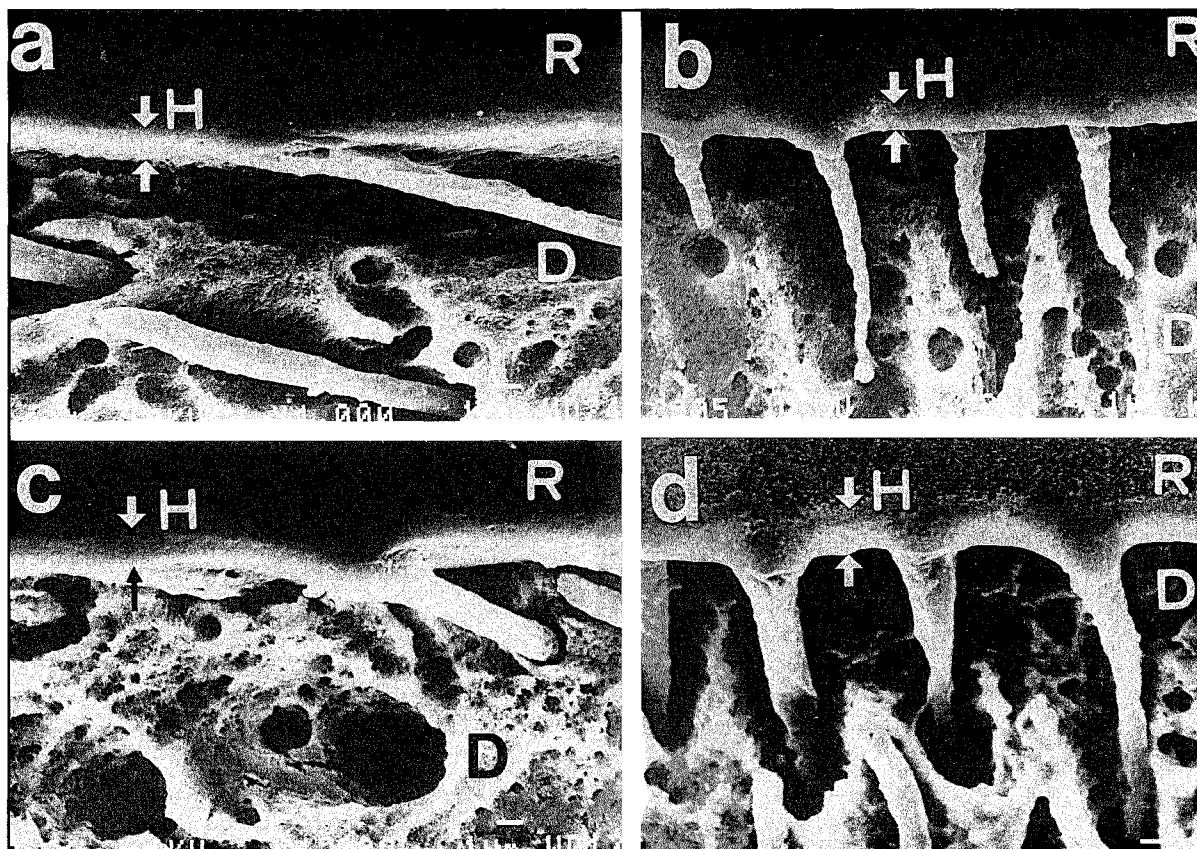


Figure 3. SEM of the Liner Bond II/dentin interface after treatment with 10% phosphoric acid and 5% sodium hypochlorite. **A&B.** Occlusal and gingival walls of the cavity when LB-primer was applied once. Thickness of the hybrid layer is approximately 1  $\mu\text{m}$  (arrows). **C&D.** Occlusal and gingival walls of the cavity when LB-primer was applied several times. Thickness of the hybrid layer is approximately 2  $\mu\text{m}$  (arrows). (magnification X3320; bar = 1  $\mu\text{m}$ .)

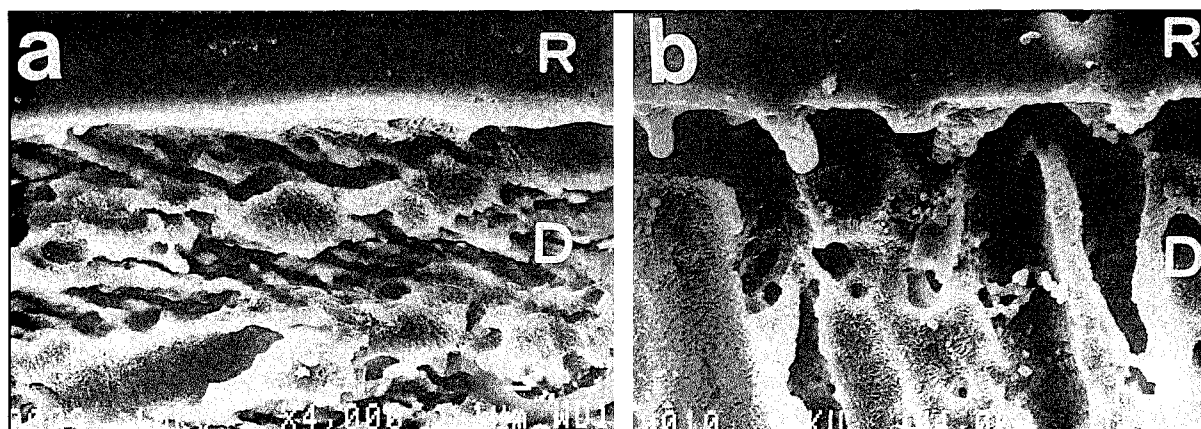


Figure 4. SEM of the Fluoro Bond/dentin interface of the Group FB(S) after treatment with 10% phosphoric acid and 5% sodium hypochlorite. **A.** Occlusal wall of the cavity. **B.** Gingival wall of the cavity. The hybrid layer is not clearly observed. (magnification X3320; bar = 1  $\mu\text{m}$ .)

concluded that FB-primer probably removed the peritubular dentin matrix more efficiently than LB-primer. According to the results of this study, the removal of the peritubular dentin matrix appeared to show no difference between LB and FB. The functional difference of each adhesive-monomer of these primers (LB: Phenyl-P, FB: 4-AET), especially the difference of the pH of both monomers (LB: 1.4, FB: 2.5—information provided by the manufacturers), may have caused this difference. Ikemura and others (1996) reported that the ionized 4-AET in water/HEMA solution would lead to sufficient chemical interaction with dentinal tissue, resulting in good penetration of bonding resin into the superficial dentin at the adhesive interface. High reactivity of 4-AET might have achieved better surface adhesion to dentin. Kanemura and others (1996) evaluated the effect of two priming times for the FB-primer on dentin bond strength. They concluded that there was no significant difference of tensile bond strength among the groups treated by FB-primer for 10 seconds or 30 seconds. Therefore, the efficacy of FB-primer may not depend on application time or frequency. The difference of viscosity of the two primer solutions might have partly influenced our result [Contact angles of each primer to human dentin were: LB— $\theta = 14^\circ$ ; FB— $\theta = 20^\circ$ ; water— $\theta = 28^\circ$  (information provided by the manufacturers)]. FB-primer is a more viscous solution than LB-primer.

Yoshiyama and others (1996) measured the regional bond strength of LB in natural and artificial wedge-shaped defects of human teeth that were extracted for periodontal reasons. Their results indicated that the tensile bond strengths of bonds made to natural lesions were significantly lower than to artificially created lesions. However, they reported no significant differences between bonds made to gingival walls and occlusal walls of natural or artificial lesions. In our study, we used sound third molars from young patients. With both adhesive systems and methods of primer application, bond strengths to gingival walls were significantly lower than those to occlusal walls. Yoshiyama and others (1996) reported that LB formed no resin tags in the tubules in the gingival site of cavities prepared in normal cervical dentin. Our results showed that the thickness of the resin-infiltrated layer was almost the same as reported by Yoshiyama and others (1996), but with tag formation within the tubules. Tagami and others (1992) reported that the permeability of old normal dentin was much lower than that of young normal dentin. The orientation of the dentinal tubules within the occlusal wall was generally parallel to the prepared surface, while those of the gingival wall were perpendicular to the interface. Thus, there were more tubules connected to the cut surface at the

gingival site than were seen at the occlusal site. Further research is needed to investigate the relationship between the direction of dentinal tubules and dentin bond strength.

In the artificially created wedge-shaped defects, the dentin was normal and the tubules were patent. This probably allowed better resin infiltration. It is unclear why the bond strength to the gingival wall decreased in our study. Presumably, the existence of water provided from the pulpal side of the dentinal tubule, due to the higher permeability of the young tooth, might have negatively affected the bond strength to the gingival wall. Several studies have demonstrated that natural cervical abraded dentin was difficult to acid etch for bonding due to hypermineralization of the surface of the dentin (Duke & Lindemuth, 1990, 1991; Harnirattisai & others, 1993; Van Meerbeek & others, 1992; Pashley & others, 1996). In this study, multiple applications of the self-etching primer were intended to supply an adequate amount of primer into a wedge-shaped defect. The priming time recommended by the manufacturer for FB-primer was very short, limiting the priming applications of FB to five. Therefore the same frequency was applied to LB-primer. With this study, it became clear that bond strength to the artificial wedge-shaped defect improved significantly by multiple applications of LB-primer. Thus, it is of crucial importance to evaluate the effect of multiple primer applications on bonding to natural wedge-shaped defects, as well as to investigate the suitable frequency for achieving maximum bond strength.

## CONCLUSIONS

By multiple primer applications of Liner Bond II, bond strength to the artificial wedge-shaped defect improved significantly. However, bond strength of Fluoro-Bond indicated no statistical difference when altering the number of primer application. With both adhesive systems and primer application methods, tensile bond strength to the gingival wall was significantly lower than to the occlusal wall. Multiple primer applications to these defects may be an effective method to treat properly the dentin surface to produce strong bonds.

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# In Vitro Microleakage of Glass-Ionomer Composite Resin Hybrid Materials

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## Clinical Relevance

Microleakage of glass-ionomer composite resin hybrid materials placed on root surface cavities did not reveal significant differences when compared to composite resin. Two polyacid-modified composite resins and a resin-modified glass ionomer showed less microleakage than a conventional glass-ionomer cement.

## SUMMARY

The purpose of this study was to evaluate the microleakage of six glass-ionomer composite resin hybrid materials compared with a glass-ionomer cement and a composite resin. Standardized class 5 dentin cavities were prepared on root surfaces of 240 extracted human teeth that were

randomly assigned to eight groups and restored using the following restorative systems: (I) Vitremer, (II) Compoglass, (III) Photac-Fil Aplicap, (IV) Variglass, (V) Dyract, (VI) Fuji II LC, (VII) Ketac-Fil Aplicap, and (VIII) Z100. The teeth were thermocycled, placed in a 2% methylene blue solution, and sectioned with diamond disks. Dye penetration was scored on a scale of 0-3. Results showed no significant differences among groups VIII, IV, I, V, VI, III, and II. There were also no significant differences among groups VI, III, II, and VII.

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## INTRODUCTION

One essential factor in the longevity of a restoration is the marginal sealing ability of the restorative material (Sim & Sidhu, 1994). Dimensional changes and lack of adaptation of the restoration to cavity walls can lead to marginal leakage (Nelsen, Wolcott & Paffenbarger, 1952), with fluid and molecular movement and the ingress of bacteria or bacterial nutrients (Mount, 1994). Long-term sealed restorations are required to prevent microleakage and its sequelae: staining, postoperative sensitivity, pulpal irritation, and recurrent caries (Bauer & Henson, 1984).

The search for a restorative material with adhesive and caries-protective properties as well as one requiring a simple clinical technique has led to the recent development of glass-ionomer composite resin hybrid materials (Gladys & others, 1997). These materials are intended to overcome the clinical problems associated with conventional glass-ionomer cements such as short working time, long setting time, and early water sensitivity (Wilson, 1989; McLean, 1992), while at the same time preserving their advantageous properties, which include adhesion to dental tissues (Hotz & others, 1977; Wilson & McLean, 1988), fluoride release (Kidd, 1978; Maldonado, Swartz & Phillips, 1978), esthetics (Mount, 1994), and thermal insulation (McLean, 1992).

In addition to the components of conventional materials, the formulas of glass-ionomer composite resin hybrid materials contain resins such as hydroxyethyl methacrylate (HEMA), which act as

photocurable monomers (Kanchanasavita, Pearson & Anstice, 1995). Resin-modified glass ionomers set by means of two mechanisms: the acid-base reaction common to all glass-ionomer cements and a photochemical polymerization of water-soluble monomers and/or pendant methacrylate groups (Wilson, 1990). Polyacid-modified composite resins contain either or both of the essential components of a glass-ionomer cement, but at levels that are insufficient to produce an acid-base cure without initiation by light (McLean, Nicholson & Wilson, 1994).

The inclusion of resins may interfere with the dimensional stability (Kanchanasavita & others, 1995) and the cavity sealing ability of these new hybrid materials. Therefore, this study was conducted to evaluate the marginal leakage of three resin-modified glass-ionomer cements and three polyacid-modified composite resins, in comparison with a glass-ionomer cement and a composite resin.

*Table 1. Restorative Systems Tested*

Group	Brand Name	Type	Batch #	Manufacturer
I	Vitremer	RMGIC	19960604 Powder: 6650 Liquid: 650 Primer: 627 Finishing gloss: 6A	3M Dental Products St Paul, MN 55144
II	Compoglass	PMCR	708535 SCA: 716109	Vivadent Ets Schaan, Liechtenstein
III	Photac-Fil Aplicap	RMGIC	21032 Ketac Conditioner: 0004 Ketac Glaze: 098	ESPE Seefeld/Oberbay, Germany
IV	Variglass	PMCR	27572 PUB3: 092691	Dentsply Ind & Com Ltda Petrópolis, Brazil 25665010
V	Dyract	PMCR	59503125 PSA: 9503145	L D Caulk/Dentspy Milford, DE 19963
VI	Fuji II LC	RMGIC	270751 Fuji Varnish: 240851	GC America Chicago, IL 60658
VII	Ketac-Fil Aplicap	GIC	Z095 Lot 0023 Ketac Conditioner: 0004 Ketac Glaze: 098	ESPE
VIII	Z100/Scotchbond Multi-Purpose Plus	CR DAS	6ID Acid primer: 6LH Adhesive: 6DE	3M Dental Products

RMGIC = resin-modified glass-ionomer cement; PMCR = polyacid-modified composite resin; GIC = glass-ionomer cement; CR = composite resin; DAS = dentin bonding system.

Table 2. Restorative Techniques Used According to Manufacturers' Recommendations

Restorative System	Dentin Pretreatment	Dispensing and Mixing	Insertion	Light Curing (8)	Surface Protection
Vitremer (1)	Vitremer Primer (1)	Powder/Liquid ratio of 2.5/1 (g/g) Manually	Centrix syringe (7)	40 seconds	Vitremer Finishing Gloss (1)
Compoglass (2)	Compoglass SCA Bonding Agent (2)	Capsule (no mixing)	Cavifil injector (2)	40 seconds	None
Photac-Fil Aplicap (3)	Ketac Conditioner (3)	Predosed capsule Capmix (3)	Aplicap system (3)	40 seconds	Ketac Glaze (3)
Variglass (4)	Prisma Universal Bond 3 (4)	Powder/Liquid ratio of 5/1 (g/g) Manually	Centrix syringe (7)	40 seconds	Prisma Universal Bond 3 Adhesive (4)
Dyract (5)	Dyract PSA Primer/Adhesive (5)	Capsule (no mixing)	Capsule tip and syringe (5)	40 seconds	None
Fuji II LC (6)	None	Powder/Liquid ratio of 3/1 (g/g) Manually	Centrix syringe (7)	20 seconds	GC Fuji Varnish (6)
Ketac-Fil Aplicap (3)	Ketac Conditioner (3)	Predosed capsule Capmix (3)	Aplicap system (3)	None	Ketac Glaze (1)
Z100 (1)/Scotchbond Multi-Purpose Plus (1)	SBMP Etchant (1) SBMP Primer (1) SBMP Adhesive (1)	None	Centrix syringe (7)	40 seconds	None

(1) = 3M Dental Products; (2) = Vivadent; (3) = ESPE; (4) = Dentsply; (5) = L D Caulk/Dentsply; (6) = GC America; (7) = Centrix Inc, Shelton, CT 06484; (8) = Visilux 2, 3M Dental Products.

## METHODS AND MATERIALS

### Experimental Design

The main factor under study was the restorative system at eight levels. The experimental units were 240 human extracted teeth, restored in 10 blocks of 24 teeth each. Each block contained three teeth restored with each material. Within each block, the order in which the eight materials were used was randomly determined. A randomized complete block design was used to systematically control the variability arising from known nuisance sources (Montgomery, 1991). The response variable was microleakage, evaluated blind and independently by three examiners using an ordinal scale from 0 to 3.

### Teeth Preparation

Two hundred and forty freshly extracted uniradicular human teeth (incisors, canines, and premolars) were stored in 2% formalin solution, cleaned, selected, and

then stored in distilled water at room temperature to prevent dehydration. All selected teeth were free of caries and restorations on their root surfaces. A standardized cylindrical class 5 cavity preparation was placed on the facial root surface, about 4 mm below the cemento-enamel junction. The cavity preparations were approximately 1.5 mm in diameter and 1.5 mm deep, made with a special diamond bur #015 (KG Sorensen, Barueri, SP, Brazil, 06454-920) in a high-speed handpiece (Dabi Atlante SA, Ribeirão Preto, SP, Brazil, 14095-000), using constant water-spray coolant. The root apices of each tooth were removed, and the surface was acid etched, primed, and sealed with an adhesive composite resin (Z100/Scotchbond Multi-Purpose, 3M Dental Products, St Paul, MN, 55144).

### Restoration

The 240 prepared teeth were randomly assigned to the 10 blocks (24 teeth each). In each block, three teeth were restored with one of the eight materials

(Table 1) according to the manufacturer's recommendations (Table 2).

Before restoration, all cavities were cleaned by air/water spray to loosen all sediment left from preparation, then lightly dried with air to avoid dentin dehydration. The restorative materials were placed in one increment, since the depths were less than 2 mm. The restored teeth were stored in a humidifier at  $37 \pm 1^\circ\text{C}$  for 24 hours and the restorations finished wet with a graded series of Sof-Lex disks (3M do Brasil Ltda, Sumaré, SP, 13001, Brazil). All preparation of teeth, restoration, and finishing were performed by one operator.

### Thermocycling Regimen and Dye Penetration

The teeth were placed into separate mesh bags and thermocycled together for 500 cycles in water between  $5 \pm 2^\circ\text{C}$  and  $55 \pm 2^\circ\text{C}$  with a dwell time of 60 seconds in each bath and a 15-second transfer time between baths. All external surfaces of each tooth were coated with two layers of nail varnish, leaving a 1.5 mm-wide margin around the restoration free of varnish. The teeth were placed in a 2% methylene blue solution for 24 hours at room temperature, rinsed under running water, then the nail varnish was removed. The teeth were sectioned in a faciolingual direction through the center of each restoration with a slow-speed diamond saw (KG Sorensen). Both halves of each sectioned tooth were evaluated blind and independently by three examiners with a stereomicroscope (Meiji Techno America, San Jose, CA 95131-2041) at X16 to determine the extent of the microleakage at the occlusal and gingival margins. The following criteria were used to score dye penetration: 0 = no dye penetration; 1 = dye penetration to 1/2 of the occlusal or gingival wall; 2 = dye penetration to full length of the occlusal or gingival wall; 3 = dye penetration including axial wall.

### Statistical Analysis

Data were analyzed by the Friedman Test (Campos, 1983). This test was chosen due to the nature of the qualitative random variable, which employs scores to evaluate the phenomenon under study (microleakage). The block design was the determining factor in the choice of the Friedman Test. Each block had three teeth using every restorative system. Each tooth was sagittally sectioned into two halves, which were scored at gingival and occlusal walls by three examiners, totalling 12 observations per tooth. The median was taken from 36 observations (2 halves x 2 walls x 3 examiners x 3 teeth) in each block. The Friedman Test first considered the rank of median values in each block, and then the sum of ranks per restorative

system. Subsequently, pairwise comparison was employed to check the hypothesis of equality among the groups of materials studied by means of the least significant difference (LSD). The coefficient of agreement among the three examiners was verified by Kappa estimator (Landis & Koch, 1977) considering every microleakage score. The statistic calculations were performed by Stata (Computing Resource Center, Santa Monica, CA, 90401) software.

## RESULTS

The microleakage median scores and ranks per restorative system in each block, the sum of rank values, and pairwise comparisons for the restorative systems are presented in Table 3. The Friedman Test showed statistically significant differences among the restorative systems at the 5% probability level ( $\chi^2 = 37.11$ ;  $\alpha < 0.05$ ). Comparisons of the sum of ranks showed no statistically significant differences among Groups I (Vitremer), II (Compoglass), III (Photac-Fil Aplicap), IV (Variglass), V (Dyract), VI (Fuji II LC), and VIII (Z100). Group VII (Ketac-Fil Aplicap) showed the highest microleakage but was not statistically different from Groups II (Compoglass), III (Photac-Fil Aplicap), and VI (Fuji II LC). The least significant difference (LSD) used to compare the sums of ranks was 34. Kappa values obtained for pairwise agreement among the examiners were 0.65, 0.69, and 0.71.

## DISCUSSION

Carious or abrasion-type lesions in the cervical areas of teeth are usually restored with either composite resin or glass-ionomer cement. Despite the improvements in bond strengths of dentin adhesives and resin-modified glass ionomers, the marginal seal of cervical restorations remains a concern (May & others, 1996).

In vitro evaluation of dye penetration is frequently used to test the sealing efficiency of restorative systems. None of the currently available dye penetration tests are ideal, and as tests are not standardized, comparison of results from different studies is difficult (Chan & Jones, 1993). As the results depend on the experimental design and the selected criteria of evaluation, reaching absolute conclusions and accurately ranking the restorative systems is difficult (Déjou, Sindres & Camps, 1996). In this study, interexaminer consistency was tested to exclude any possible form of bias. Kappa values obtained for pairwise agreement were considered "substantial," according to Landis and Koch (1977). These findings support the reliability of the evaluation criteria used in this study.

The results of this study showed that resin-

Table 3. Spreadsheet Used to Calculate the Friedman Test, Showing the Microleakage Median Scores and Ranks Per Restorative System, the Sum of Ranks and Pairwise Comparison among Groups, by Means of the Least Significant Difference

Restorative System	Block	1	2	3	4	5	6	7	8	9	10	Sum of Ranks*
Z100/ SBMP Plus	Median score	0.0	0.0	0.5	0.0	0.0	0.0	0.0	0.0	0.5	0.0	18.0 <sup>a</sup>
	Rank	1.0	2.5	2.0	1.5	1.0	1.5	1.5	2.0	3.0	2.0	
Variglass	Median score	1.5	0.0	0.0	0.0	1.0	0.0	1.0	1.0	0.0	0.0	31.0 <sup>a</sup>
	Rank	7.0	2.5	1.0	1.5	4.0	1.5	4.5	5.5	1.5	2.0	
Vitremer	Median score	1.0	0.0	1.0	1.5	1.0	0.5	1.0	0.0	0.0	0.5	37.0 <sup>a</sup>
	Rank	4.5	2.5	3.5	6.0	4.0	3.0	4.5	2.0	1.5	5.5	
Dyract	Median score	0.5	0.0	1.0	1.0	2.5	1.0	0.0	1.0	1.5	0.5	44.5 <sup>a</sup>
	Rank	2.0	2.5	3.5	4.0	7.5	5.5	1.5	5.5	7.0	5.5	
Fuji II LC	Median score	1.0	1.0	1.5	1.0	1.0	1.0	1.0	1.0	1.0	0.5	49.5 <sup>ab</sup>
	Rank	4.5	5.0	6.0	4.0	4.0	5.5	4.5	5.5	5.0	5.5	
Photac-Fil Aplicap	Median score	1.0	1.5	1.5	1.0	0.5	1.5	2.0	0.0	1.0	0.5	50.0 <sup>ab</sup>
	Rank	4.5	7.0	6.0	4.0	2.0	7.0	7.0	2.0	5.0	5.5	
Compoglass	Median score	1.0	1.3	1.5	3.0	2.0	0.8	1.0	1.0	1.0	0.0	51.0 <sup>ab</sup>
	Rank	4.5	6.0	6.0	7.5	6.0	4.0	4.5	5.5	5.0	2.0	
Ketac-Fil Aplicap	Median score	3.0	2.5	2.5	3.0	2.5	3.0	3.0	3.0	3.0	2.5	79.0 <sup>b</sup>
	Rank	8.0	8.0	8.0	7.5	7.5	8.0	8.0	8.0	8.0	8.0	

\*Values followed by the same letter did not differ from each other. Least significant difference (lsd) = 34. Probability level of 5% ( $\alpha = 0.05$ ). Median scores were taken from 36 observations (2 halves x 2 walls x 3 examiners x 3 teeth).

modified glass-ionomer cements and polyacid-modified composite resins had microleakage patterns similar to a composite resin, but statistically different from a conventional glass-ionomer cement. However, some hybrid materials tested (Fuji II LC, Photac-Fil Aplicap, and Compoglass) were not significantly different from a glass-ionomer cement that showed the higher microleakage median score.

Glass-ionomer cement requires a prolonged maturation time and should not be challenged with dehydration within 6 months of placement (Mount, 1990). Preparation of the specimens for this investigation may have led to some dehydration of the restorations, especially during application/drying of the nail varnish. Bouschlicher, Vargas, and Denehy (1996) showed that inadvertently desiccating

the restoration prior to dye immersion increased microleakage scores with some resin-modified glass ionomers, glass-ionomer cements, and microfill resins. If contraction under desiccating conditions disrupts the bond at the cervical margin or is far greater than the expansion by water absorption (Wilson & Paddon, 1993), there could be a resultant increase in microleakage (Bouschlicher & others, 1996). It should be remembered that some of the materials evaluated in this study were likely to be more technique sensitive than others, perhaps leading to greater differences.

The resin-modified glass-ionomer cements Fuji II LC, Photac-Fil Aplicap, and Vitremer contain polymerizable monomers such as 2-hydroxyethyl methacrylate (HEMA) in their composition (Gladys



& others, 1997). In Fuji II LC and Photac-Fil Aplicap, these monomers are merely blended with a polyalkenoic acid liquid; in Vitremer, besides a simple mixture of HEMA with a polyalkenoic acid, the latter itself is also modified by the attachment of polymerizable methacrylate side groups (Gladys & others, 1997). In addition, Vitremer uses a primer that improves the wetting of dentin (Friedl, Powers & Hiller, 1995). Its composition resembles Scotchbond Multi-Purpose Primer, which contains polyalkenoic copolymer, and HEMA (Vargas, Fortin & Swift, 1995). On the basis of these chemical characteristics and physical properties reported in the literature (Gladys & others, 1997; Burgess, Norling & Summitt, 1994), it seems that Fuji II LC and Photac-Fil Aplicap more closely approximate conventional glass ionomers, which could explain why, in this study, these materials had microleakage patterns similar to Ketac-Fil Aplicap, while Vitremer provided different results.

The properties of polyacid-modified composite resins are closer to those of composite resins (Gladys & others, 1997). In this study, Dyract and Variglass showed microleakage scores not statistically different from Z100 used with Scotchbond Multi-Purpose Plus. Polyacid-modified composite resins exhibit little of the inherent glass-ionomer adhesion to dentin and require dentinal bonding agents to obtain meaningful adhesion (Burgess & others, 1994). Dyract uses dentin pretreatment with Dyract PSA, a primer containing adhesion-promoting monomer (PENTA, TEGMA), in an elastomeric resin and acetone solvent (Abdalla, Alhadainy & Garcia-Godoy, 1997). It is assumed that the hydrophilic phosphate groups in the primer form an ionic bond with the calcium of tooth hydroxyapatite (Yap, Lim & Neo, 1995). Variglass primer is 6% PENTA (phosphonated penta-acrylate ester) and 30% HEMA in an ethanol solution (Friedl & others, 1995). It is a weakly acidic, self-etching primer that promotes adhesion. SEM images suggest that a micromechanical interlocking may be present at the interface of Variglass and pretreated dentin, revealing a hybrid layer similar to that observed with composite resins; however, it was nonuniform and thinner (Carvalho & others, 1995). Improved bond strengths because of mechanical and chemical bonding mechanisms could be attributed to the microleakage performance of these materials.

It is unclear why, in spite of the use of a Compoglass SCA bonding agent, one polyacid-modified composite resin (Compoglass) presented microleakage that was not statistically different from Ketac-Fil Aplicap, while the others (Dyract and Variglass) were.

New products have been marketed aggressively without sufficient data concerning their physical

properties (Sim & Sidhu, 1994). This study, based on the laboratory performance of three resin-modified glass-ionomer materials and three polyacid-modified composite resins, provided further information on these products. However, hybrid restorative materials show a pronounced diversity in their properties, which may indicate that an optimum composition has yet to be found (Gladys & others, 1997).

## CONCLUSIONS

Under the experimental conditions used in this study, the microleakage of resin-modified glass ionomers (Vitremer, Fuji II LC, and Photac-Fil Aplicap) and polyacid-modified composite resins (Variglass, Dyract, and Compoglass) were not significantly different from the composite resin Z100. Variglass, Vitremer, and Dyract showed significantly less microleakage than the glass-ionomer cement. Fuji II LC, Photac-Fil Aplicap, and Compoglass did not differ from the glass-ionomer cement, Ketac-Fil Aplicap.

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# The Effect of Surface Treatment on the Bond Strength of Resin Composite to Dentin

Y BENDERLI • T YÜCEL

## Clinical Relevance

Etching of dentin with a strong acid does not always lead to strong bonding.

## SUMMARY

The purpose of this study was to investigate the effect of various dentin acid treatments on the tensile bond strength of resin composite to dentin, mediated by Prisma Universal Bond 2. Acid solutions (maleic acid, Na-EDTA, phosphoric acid, citric acid/ferric chloride) were applied to dentin surfaces for 15 seconds or 60 seconds, and tensile bond strengths evaluated. For all acids, there was a significant difference in bond strength for the two treatment times; for phosphoric acid and citric acid/ferric chloride, the 15-second application resulted in a higher bond strength, while the reverse was true for Na-EDTA (sodium-EDTA) and maleic acid. The highest bond strength (19.6 MPa) was

associated with 15-second citric acid/ferric chloride application, and the lowest bond strength (5.6 MPa) with 60-second application of citric acid/ferric chloride. The bond strengths with 60-second citric acid/ferric chloride (5.6 MPa), 15-second Na-EDTA (5.8 MPa), and 60-second phosphoric acid (6.3 MPa) were not significantly different from the control (5.7 MPa).

## INTRODUCTION

Ideal restorative materials will bond strongly to tooth tissues, will possess physical properties similar to natural teeth, will be easy to apply, and will be aesthetically favorable. The resin-composite materials have been developed in such a way that they can bond to enamel and dentin chemically and/or micromechanically (Burke, 1991; Eick & others, 1991, 1992, 1993; Leinfelder, 1993; Willems & others, 1993). The acid-etching technique was a significant step forward towards providing a strong bond between resin composite and enamel (Buonocore, 1955; Buonocore, Wileman & Brudevold, 1956). This technique primarily provided micromechanical bonding, but also acted as a stimulus for research on the

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surface preparation of dentin (Eick & others, 1991, 1992, 1993; Berry, von der Lehr & Herrin, 1987; Bitter, 1989, 1990; Brännström, Glantz & Nordenvall, 1979; Finger, Manabe & Alker, 1989; Gendusa, 1994; Krejci & others, 1990; Kurosaki & others, 1990).

The first dentin bonding agents were designed to bond chemically to some component of the dentin. However, it became apparent that bonding to the smear layer was weak, and subsequently failed cohesively. When compared to the bonding of resin composite to enamel, dentin bonding exhibited lower bond strength values (Burke, 1991; Buonocore & others, 1956; Marshall, 1993; Retief & others, 1988), and

than 3 weeks prior to use. The teeth were sectioned horizontally into thirds (Figure 1a-c). Approximately 3 mm-thick disk-shaped slices were obtained as the middle third from each tooth by use of a diamond disk revolving at low speed under running water. These disk-shaped slices were divided into four pie-shaped and equal pieces (Figure 1d,c) and a circle-shaped figure (5 mm in diameter) was drawn on each dentin section (Figure 1f). Following this procedure, a piece of dentin (3 mm in height and 5 mm in diameter) was obtained from each section of the tooth (Figure 1g). One end of each dentin section (1 mm height) was adapted to the narrow orifice of a

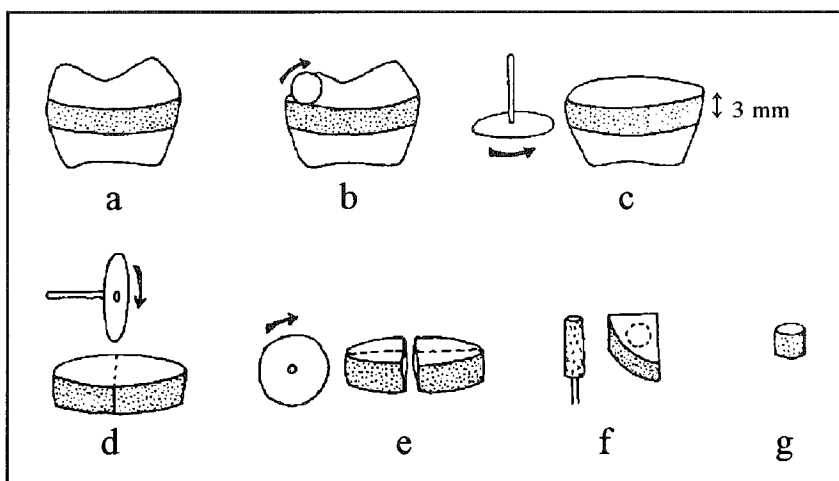


Figure 1. Preparation of dentin surfaces

it was clear that smear layer removal was necessary as a preliminary step. For this purpose, contemporary research is primarily concentrated on the preparation of the dentin to remove the smear layer and surface minerals, but without causing irritation to the dental pulp. The remaining collagen can then be impregnated by a suitable hydrophilic monomer, which will polymerize to form the so-called "hybrid" or "resin-reinforced" layer (Eick & others, 1993; Berry & others, 1987; Bitter, 1989, 1990; Finger & others, 1989; Gendusa, 1994; Krejci & others, 1990; Kurosaki & others, 1990; Davis & others, 1992; Heymann & Bayne, 1993; McInnes-Ledoux, Austin & Cleaton-Jones 1984; Meryon, Tobias & Jakeman, 1987; Pashley, 1992; Pashley & others, 1993).

The purpose of this study was to compare four acids, with two application times each, for their effect on the tensile bond strength to dentin of a resin composite mediated by a proprietary dentin bonding agent.

## METHODS AND MATERIALS

Unerrupted human third molars were extracted and stored at 4 °C in isotonic saline solution for no longer

than 3 weeks prior to use. The teeth were sectioned horizontally into thirds (Figure 1a-c). Approximately 3 mm-thick disk-shaped slices were obtained as the middle third from each tooth by use of a diamond disk revolving at low speed under running water. These disk-shaped slices were divided into four pie-shaped and equal pieces (Figure 1d,c) and a circle-shaped figure (5 mm in diameter) was drawn on each dentin section (Figure 1f). Following this procedure, a piece of dentin (3 mm in height and 5 mm in diameter) was obtained from each section of the tooth (Figure 1g). One end of each dentin section (1 mm height) was adapted to the narrow orifice of a tapered mold (Figure 2) by use of utility wax. Then each dentin piece was mounted in self-cured acrylic resin, in tapered molds, 7 mm high x 5 mm in internal diameter at the base x 10 mm in internal diameter at the top (Figure 2). Six samples were prepared in each group.

Four acids were applied passively to each exposed dentin surface for two different time periods (15 and 60 seconds). Following the acid application, all samples were washed and dried, a dentin bonding agent (Prisma Universal Bond 2, L D Caulk/Dentsply, Milford, DE 19963) placed and blown thin, and polymerized by a light source (Heliolux Type HL 1 F Nr 120628, Vivadent, Schaan, Liechtenstein) for 20 seconds. Plexiglass molds (the same size as

the tapered molds) were placed on the dentin slices (Figure 2) and filled with 3 x 1.5 mm-thick layers of hybrid posterior composite (Prismatic Ful-Fil, Caulk/Dentsply) and cured for 40 seconds. The composite was in contact only with the dentin surface (Figure 2). All materials used in this study are shown in

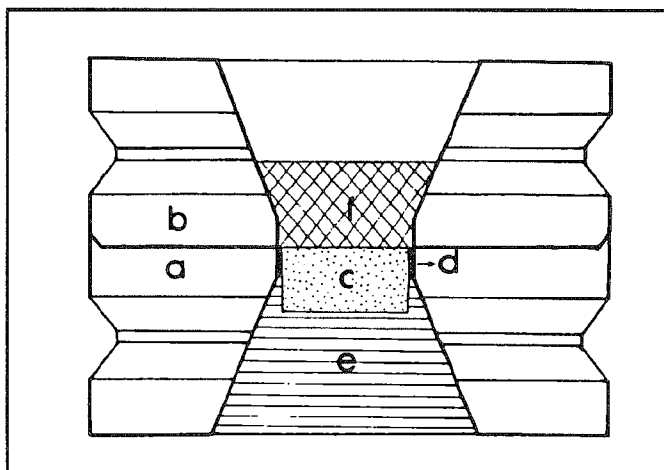


Figure 2. Dentin sections in tapered molds; a = brass mold; b = plexiglass mold; c = dentin section; d = resin utility wax; e = acrylic resin; f = composite.

Table 1. Materials Used in This Study

	Materials	Composition	Manufacturer	Batch #
Composite	Prisma Ful-Fil	BIS-GMA barium glass colloidal silver	L D Caulk/Dentsply Milford, DE 19963	0402881 U - B62
Bonding Agent	Prisma Universal Bond 2	PENTA phosphate ester of BIS-GMA glutaraldehyde	L D Caulk/Dentsply	121190
Etchant	Phosphoric Maleic Na-EDTA Citric acid/ Ferric chloride		prepared in laboratory conditions	

Table 1. The procedure was performed at  $25 \pm 2^\circ\text{C}$  and 50% humidity. The specimens were placed in distilled water at  $37^\circ\text{C}$  for 1 week. The same procedures were used for the control group, with the exception of acid application.

After the storage period, the samples were attached to a Universal Mechanical Testing Machine (Instron 1115 Universal Mechanical Testing Machine, High Wycombe, Bucks, England), and the breaking force (kgf) of the resin composite to the dentin was measured at a crosshead speed of 1.0 mm per minute. Mean bond strengths were calculated in MPa.

The groups means were compared by the Mann Whitney U test ( $P < 0.05$ ).

## RESULTS

The mean bond strengths are shown in Table 2 and the statistical analysis of the groups are presented in Table 3 and Figures 3 and 4.

For the 15-second application time, all bond strengths were significantly different (Table 3). The longer application time was associated with higher bond strengths for maleic acid and NaEDTA, but lower bond strengths for phosphoric acid and citric acid/ferric chloride (Table 2). The bond strength values obtained with 60-second phosphoric acid, 60-second citric acid/ferric chloride, and 15-second Na-EDTA were not significantly different from the control (Figure 3).

For all acids, there was a significant difference between the bond strengths for the 15-second and 60-second applications (Table 3). Citric acid/ferric chloride was associated with the highest bond strength (19.6 MPa), and Na-EDTA with the lowest bond strength (5.8 MPa). For the 60-second

application, the bond strength associated with maleic acid was the highest (14.9 MPa), and with citric acid/ferric chloride the lowest (5.6 MPa).

## DISCUSSION

The main reason for the selection of the demineralizing solutions in this study was that two of them are weak (maleic acid and Na-EDTA) and the other two are strong (phosphoric acid and citric acid/ferric chloride) (Windholz, 1988). The pH values of the acids used in this study were as follows: maleic acid pH = 2.3; Na-EDTA pH = 5.2; citric acid/ferric chloride pH = 0.9; phosphoric acid pH = 0.1 (Windholz, 1988). In order to eliminate the variable of the dentin

adhesive on the bond strength, one type of adhesive (Prisma Universal Bond 2; PUB 2), was used. Since the purpose of this study was to evaluate only the effect of various acids on the dentin surface, maleic acid, Na-EDTA, citric acid/ferric chloride, and phosphoric acid solutions were applied to dentin before the PUB 2 adhesive was applied.

In previous studies, EDTA was found to be very effective in dentin treatment (Eick & others, 1991; Finger & others, 1989); however, it has been noted by various investigators that it may also cause microleakage problems because of over-widening the dentinal tubules

Table 2. Acids Used and Treatment Times with Resultant Bond Strengths (MPa)

Group	Acid	Concentration Percent	Treatment Times	Bond Strength	$\pm$ SD (MPa)
1	Phosphoric	37	15	12.9	2.75
2	Phosphoric	37	60	6.3	1.46
3	Maleic	10	15	9.0	1.50
4	Maleic	10	60	14.9	1.54
5	Na-EDTA	10	15	5.8	0.58
6	Na-EDTA	10	60	6.9	1.29
7	Citric acid/ Ferric chloride	10/3	15	19.6	3.79
8	Citric acid/ Ferric chloride	10/3	60	5.6	1.18
9	Control			5.7	1.32

Table 3. Statistical Evaluation of Bond Strengths between Composite Resin and Dentin Tissue

	Comparing Groups		Statistical Analysis
	A	B	
Part I (Column A: 15 seconds; Column B: 60 seconds)	MA	MA	$P < 0.05$
	FC/CA	FC/CA	$P < 0.05$
	Na-EDTA	Na-EDTA	$P < 0.05$
	PA	PA	$P < 0.05$
Part II (Both Columns A & B 15 seconds)	PA	FC/CA	$P < 0.05$
	MA	Na-EDTA	$P < 0.05$
	PA	Na-EDTA	$P < 0.05$
	MA	FC/CA	$P < 0.05$
	FC/CA	Na-EDTA	$P < 0.05$
	PA	MA	$P < 0.05$
Part III (Both Columns A & B 60 seconds)	PA	FC/CA	NS
	MA	Na-EDTA	$P < 0.05$
	PA	Na-EDTA	NS
	MA	FC/CA	$P < 0.05$
	FC/CA	Na-EDTA	$P < 0.05$
	PA	MA	$P < 0.05$

MA = maleic acid; PA = phosphoric acid; Na-EDTA = sodium-EDTA; FC/CA = ferric chloride/citric acid; NS = no significant difference.

(Meryon & others, 1987). Therefore, in this study Na-EDTA solution was used instead of EDTA because it is a weak acid. It has been shown that Na-EDTA does not widen the tubules as much as EDTA (Eick & others, 1991; Finger & others, 1989; Benderli & Yücel, 1995).

When the groups in which dentin surface preparation was performed were compared with the control group, it was found that the dentin treatments improved the bond strength between the composite resin and the dentin. Thus, the results of this study are in agreement with previous studies (Finger & others, 1989; Davis & others, 1992).

Davis and others (1992) comparatively evaluated the effect of a 60-second application of 40% polyacrylic acid and a 15-second application of 10% phosphoric acid on the removal of the smear layer and the composite resin-dentin bond strength. In their study, they used Mirage Bond (Chameleon Dental Products, Kansas City, KS 66101), All-Bond (Bisco, Itasca, IL 60143), Prisma Universal Bond 2 (L D Caulk), and Scotchbond 2 (3M Dental Product, St Paul, MN 55144) adhesives. For each dentin bonding agent (adhesive), specimens were divided into three smear layer treatment groups: conditioning according to the manufacturers' instructions, removal with phosphoric acid, and removal with polyacrylic acid. When they used conditioning of dentin according to the manufacturers' instructions, these shear bond strength values were obtained: 2.93 MPa for Scotchbond 2, 3.35 MPa for Prisma Universal Bond 2, 10.78 MPa for All-Bond, and 10.86 MPa for Mirage Bond. On the other hand, using phosphoric acid for the surface treatment gave the following results: 4 MPa for Scotchbond 2, 5 MPa for Prisma Universal Bond 2, 17 MPa for All-Bond, and 14 MPa for Mirage Bond. After using polyacrylic acid for the same purpose, the following results were obtained: 1.5 MPa for Scotchbond 2, 2.5 MPa for Prisma Universal Bond 2, 3.5 MPa for All-Bond, and 6.5 MPa for Mirage Bond. They reported that phosphoric acid and polyacrylic

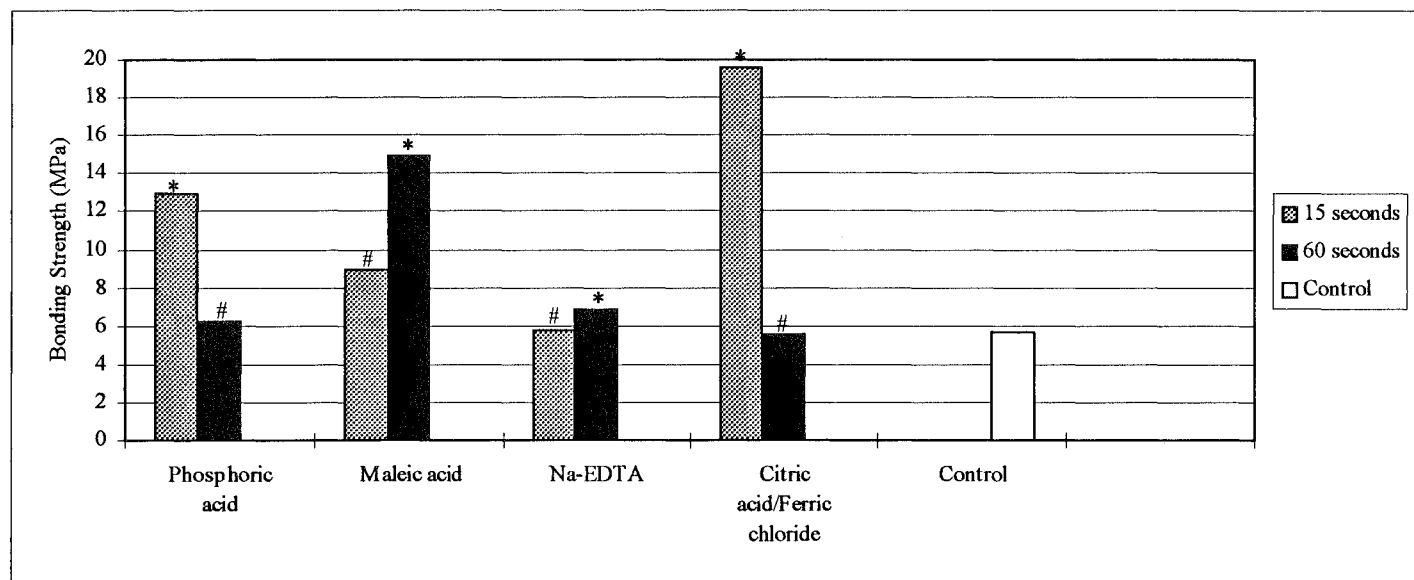


Figure 3. Comparison of bond strengths after 15-second and 60-second applications; \* = significant difference between 15- and 60-second groups; # = no significant difference from control group.



acid solutions removed the smear layer, but that phosphoric acid was associated with stronger adhesion than polyacrylic acid. In our study, it was also determined that the weakest acid (Na-EDTA) exhibited lower bond strengths for 15 or 60 seconds than the strongest acid (phosphoric acid applied 15 seconds). These results were compatible with the findings of Davis and others. In addition to this, according to Davis and others (1992) for some adhesives, i.e., All-Bond and Prisma Universal Bond 2, surface treatment of dentin using phosphoric acid gave higher bond strength values than did conditioning according to the manufacturers' instructions.

Finger and others (1989) prepared dentin surfaces of six SiC preparations using abrasive particles of various dimensions and two etching agents: Clearfil (10% citric acid + 20%  $\text{CaCl}_2$ ) for 60 seconds and Gluma 2 Cleanser (17% EDTA) for 60 seconds. Following that, dentin surfaces were evaluated either covered by smear layer (controls), or after pretreatment with cleansing agents (experimental samples). They reported that there was statistically no difference between the tensile bond strength means of the groups covered by a smear layer and the groups without a smear layer for one material (Scotchbond-Silux), but for other materials (Gluma-Lumifor, Scotchbond 2-Valux, Tenure-Silux, Clearfil-Clearfil Ray), it was found that dentin treatment improved the tensile bond strength.

In this study, when the bond strength values obtained after the application of acids for 60 seconds were compared, the results were found to be statistically different between all groups except two (Figure 4). No significant difference was found

between the phosphoric acid (60 seconds) and ferric chloride/citric acid (60 seconds) groups, and the phosphoric acid (60 seconds) and Na-EDTA (60 seconds) groups (Figure 4). Strong acids, like phosphoric acid and ferric chloride/citric acid solutions applied for 60 seconds, resulted in weaker adhesion than a 15-second application of the same acids. In a previous study (Benderli & Yücel, 1995), similar dentin surfaces were obtained using the application of phosphoric acid or ferric chloride/citric acid for 60 seconds. The appearance of these dentin surfaces looked like the one described as the "fourth score" (smear layer completely removed, peritubular dentin removed resulting in increased size of tubular orifices) of the scoring system reported by Brännström and others (1979).

When the applications of acids were evaluated comparing different time periods, phosphoric acid (strong acid) application for 60 seconds caused weaker adhesion than its application for 15 seconds. On the other hand, Na-EDTA (weak acid) application for 60 seconds resulted in a stronger bond compared to the 15-second application. The highest value of Na-EDTA obtained by using 60 seconds and the lowest value of phosphoric acid application for 60 seconds were not statistically significant (Table 3). This result is important for the success of bonding. Therefore, both application time and pH are important considerations for understanding the decalcification effect of acid on dentin tissue.

Two weak acids (maleic acid and Na-EDTA) provided a stronger bond with increased treatment time, because they cannot sufficiently remove the smear layer after 15 seconds.

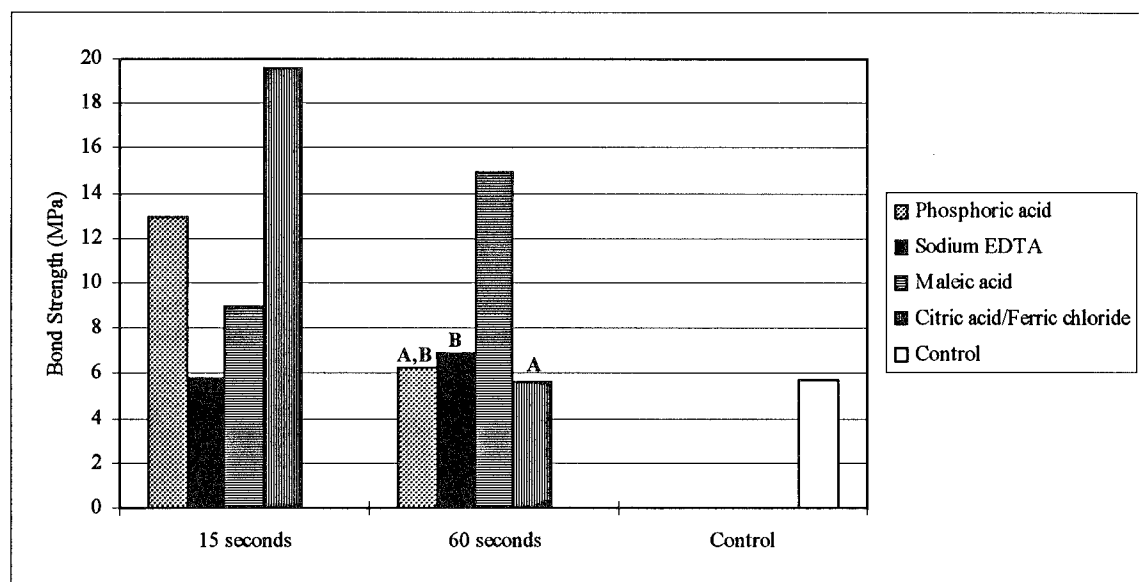


Figure 4. Comparison of bond strengths as a factor of pretreatment acid; columns with same letter are not significantly different from each other.

On the other hand, the two strong acids (phosphoric acid and citric acid/ferric chloride) exhibited a weaker bond with increased treatment time, because they could effectively remove the smear layer after 15 seconds. Used for a long time (60 seconds), they removed all the smear layer and most of the peritubular dentin. When this happens orifices of tubules are opened excessively. It may be that humidity and protein contents coming from widened tubules affect the dentin surfaces, causing decreased bonding strengths to the composite material. Pashley and others (1993) found that the adhesive material infiltrated the collagen fibrils, after removal of the mineral phase of dentin by acids and chelating agents, and this process contributed to the overall adhesion. Because of this, the bond strengths from these treatments were not significantly different from the control.

The results of our study determined that the best adhesion was obtained by the application of maleic acid for 60 seconds, phosphoric acid for 15 seconds, and ferric chloride/citric acid for 15 seconds. SEM results of a previous study (Benderli & Yücel, 1995) showed that the application of maleic acid (60 seconds), phosphoric acid (15 seconds), and ferric chloride/citric acid (15 seconds) solutions showed similar dentin surface appearances. The similarity of the bond strength values may be closely related to the similarity of surface cleanliness. The surfaces that provided the best adhesion complied with the second score (smear layer partly removed, orifices of most tubules open or partially open) or the third score (smear layer mainly removed, most tubules completely open) as described by Brännström and others (1979). When the surfaces with lower adhesion values were evaluated, it was found that the results complied with either the first score, for weak acids applied for 15 seconds, or the fourth score, for strong acids applied for 60 seconds.

### CONCLUSIONS

1. The application of the strong acids (ferric chloride/citric acid or phosphoric acid) to a dentin surface for 15 seconds gave significantly higher bond strength values than for 60-second applications.

2. Applying weak acids (maleic acid or Na-EDTA) to dentin for 15 seconds significantly decreased the bond strength values compared with 60-second applications.

3. The highest value of the weakest acid (Na-EDTA) and the lowest value of the strongest acid (phosphoric acid) for the same period of time (60 seconds) were not significantly different.

4. The highest bond strength values were obtained with the application of maleic acid (60 seconds) or phosphoric acid (15 seconds) or ferric chloride/citric acid (15 seconds).

5. Increased application time resulted in significantly improved bond strengths for weak acids but significantly weak bond strengths for strong acids.

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# Surface Treatment of Mercury-free Alloys

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## Clinical Relevance

The mercury-free metal restorative materials, gallium alloy and consolidated silver, can be polished with instruments and techniques similar to those used for conventional amalgam.

## SUMMARY

Finishing and polishing methods were examined for two metallic direct restorative materials being proposed as possible alternatives to amalgam, namely a gallium alloy and a consolidated silver alloy. The polished surfaces were compared to a conventional spherical amalgam (Tytin). After

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initial surface treatment with a 12-fluted tungsten carbide bur in a high-speed dental hand-piece, three polishing methods were evaluated: slow-speed polishing burs, rubber polishing points, and polishing disks (Sof-Lex). Each of these methods was followed by an additional surface treatment in which a pumice-flour/water slurry was applied with a rotary brush and a final surface treatment with a zinc-oxide/ethanol slurry that was applied with rotary rubber cups. The surface roughness was evaluated by profilometric measurements and light microscopy. The results showed that the smoothest surfaces for all metals were achieved with rotary finishing and polishing disks. Using the rubber points resulted in surfaces that were statistically similar to the disk-polished surfaces on all three materials. The polished surface of gallium alloy was consistently slightly rougher than that of amalgam. The consolidated silver also presented a consistently rougher surface than did amalgam, although these differences were not statistically significant. The additional polishing with pumice and zinc oxide improved the luster, but did not significantly improve the measured surface smoothness in any of the restorative materials studied.

## INTRODUCTION

Amalgam has been used for dental restorations for more than 150 years. Its advantages as a low-cost,

efficient, convenient, and reliable material have been well established, but the major disadvantage, the concern of some regarding the mercury component of amalgam, has raised the need for alternative restorative materials. Composite resins may serve as one possible alternative material, but also present some limitations, such as shrinkage upon polymerization, high thermal expansion and contraction during service, and difficult handling properties. These limitations result in compromised quality and low durability when compared to metal or ceramic restorations. Metallic materials present many advantageous physical properties such as higher compressive strength, higher tensile and flexural strength, greater toughness, and wear resistance. The need for a metallic alternative for amalgam that provides the same properties of low-cost, convenience, and efficiency for posterior restorations resulted in the development of gallium alloys (Puttkamer, 1928; Waterstrat, 1969). This material, however, had several clinical limitations such as early expansion, which resulted in postoperative pain (Navarro & others, 1996), difficult manipulation (Mash & others, 1993), margin deterioration, and surface corrosion (Navarro & others, 1996).

In 1994 another metal alternative was developed (Dariel, Admon & Lashmore, 1994), which consisted of silver particles suspended in a dilute acid solution. The acid treatment cleaned the surface by removing the oxides or other adsorbed layers from the silver particles, thus enabling their consolidation and cold welding. This acid-assisted consolidation took place at room temperature under moderate pressure. The physical properties examined showed higher rupture strength than amalgam; however, the compressive strength and hardness values were lower than those found for amalgam (Dariel, Lashmore & Ratzker, 1995). Although not currently available commercially, this material has undergone laboratory evaluation of microleakage, wear, creep, hardness and corrosion, and several manuscripts describing favorable results are currently being written or are in press. Laboratory and animal studies of biocompatibility are expected to be completed within the next year, opening the possibility for beginning human trials within 2 years. If these studies provide positive results, the material could become commercially available after completion of the human-use trials.

The clinical parameters for success of a dental restorative material are good handling properties and clinical performance. Consolidated silver is a cold-welded system that requires rotary instruments for contouring and finishing. In the present study several different established clinical techniques for contouring and polishing amalgam were applied to gallium alloy and consolidated silver, and the relative surface smoothness of the three different materials was

compared. Accurate profilometry requires that measurements be made on relatively flat surfaces, even though clinical surfaces would have rounded contours. The relative smoothness of the two surfaces, however, would be expected to be similar. The objectives of this study were to determine which clinical techniques would result in surface smoothness on the consolidated silver comparable to that achieved on gallium alloy and amalgam, and which technique would result in the smoothest surfaces overall.

## METHODS AND MATERIALS

The materials examined in the present work were consolidated silver (National Institute of Standards and Technology [NIST], Gaithersburg, MD 19102), gallium alloy (Galloy, SDI, Bayswater, Victoria, Australia 3153), and amalgam: high-copper spherical amalgam alloy (Tytin, Kerr Corp, Orange, CA 92867).

Cavities were prepared in ceramic blocks (Macor, Corning Glass, Corning, NY 14831) by machining a cylindrical hole 5 mm in diameter and 2 mm deep, 2 mm from the edge of the block. The ceramic block was 20 mm x 20 mm x 10 mm with preparations placed on three of the 10 mm x 10 mm faces of each of the blocks. All three restorative materials were placed in separate preparations on each ceramic block. A total of 43 blocks were filled, resulting in 129 samples. Filling of the preparations was performed according to the following procedures.

### Consolidated Silver Alloy

Approximately 0.6 g of silver powder (NIST laboratory batch CJ161) was immersed in 350 mL of an aqueous 0.045 mass fraction  $\text{HBF}_4$  (hydrofluoroboric acid), stirred for 1 minute, and, after the metal powder was allowed to settle for 4 minutes, the liquid was decanted, leaving the wet powder slurry. To this slurry 500 mL of an aqueous solution of  $\text{HBF}_4$ , mass fraction = 0.009, was added and stirred for several seconds followed by 1 minute of settling. The solution was decanted, and the wet slurry was carried with an amalgam carrier to the cavity and incrementally condensed in approximately six increments using a 1.25 mm-in-diameter serrated amalgam condenser (DE 671 Aesculap, Tulengen, Germany). The condensation was performed on an electronic scale (Mettler PJ 3000, Anaheim, CA 92805) at a condensation load of 2.0 kg to 2.5 kg. Condensation was done by stepping the condenser over the entire surface of each increment, taking a total of approximately 15 minutes to fill the cavity. The cavities were slightly overfilled, and the final surface was burnished with a 2.5 mm-in-diameter round dental burnisher.

## Gallium Alloy

Single-spill capsules of the commercial gallium alloy were triturated according to the manufacturer's recommendations in an amalgamator (Silamat, Vivadent, Schaan, Liechtenstein) at a medium setting for 8 seconds. The alloy was inserted into the cavity preparation and condensed with the same type of condenser as was used for the consolidated silver at a load of 1.1 kg to 1.3 kg. Four increments were placed to overfill the cavity, and excess was removed using an amalgam carver followed by burnishing with a 25 mm-in-diameter round burnisher.

## Amalgam

Single-spill capsules of Tytin amalgam were triturated in the same amalgamator at a medium setting for 11 seconds according to the manufacturer's instructions. The cavity was then filled, carved, and burnished using the same methods as described for the gallium alloy.

## Finishing Procedures

All specimens were stored for a minimum of 24 hours at 37 °C before any finishing procedure was begun. The initial surface preparation for all specimens was performed with a 12-fluted finishing bur (SS White, Lakewood, NJ 08701) at 300,000 rpm. A high-speed, water-cooled handpiece was mounted and stabilized on a fixed stand. The specimen block was fixed to a mobile machine table below the handpiece and moved under the spinning bur by manually translating the table at an even speed with a hand-controlled wheel. Excess filling material was removed until the filling was flush with the surrounding ceramic. Following this, the specimens were randomly assigned to three groups: slow-speed bur, polishing disks, and rubber points. All procedures were carried out by the same operator and at every stage of finishing, one specimen was set aside with no further treatment for microscopic examination. The methods used were as follows:

### *Finishing Burs*

Flame-shaped finishing burs (Komet, Brassier GmbH and Co, KG Lemgo, Germany) were mounted on a slow-speed handpiece at 30,000 rpm and were used to polish 42 specimens (14 of each material). The polishing was done with the hand-held handpiece lightly contacting the surface for 60 seconds. One specimen was removed from each material for microscopy, and the remaining 13 received additional polishing with a water slurry made from flour of pumice that was applied with a rotary brush mounted on the same hand-held slow-speed hand-

piece. The slurry was lightly brushed onto the surface for 20 seconds. A specimen of each material was again removed for microscopy, and the remaining 12 were given a final polish with a slurry of zinc-oxide powder immersed in ethanol. This was manually applied to the surface for 20 seconds with a rotary rubber cup mounted on the slow-speed handpiece.

### *Polishing Disks*

Finishing and polishing disks (Sof-Lex, 3M Dental Products, St Paul, MN 55144) were mounted on a slow-speed handpiece. Finishing was manually performed with each of the series of four successive abrasive grits for 15 seconds each. Fourteen specimens of each material were polished using this method, and one of each was set aside for microscopy. The second stage of polishing was performed with a slurry of pumice powder applied on a brush (as described above). The final polishing was performed with zinc oxide in ethanol, applied with a rubber cup (as described above).

### *Rubber Finishing Points*

Rubber points (Komet, Brasseler) were mounted on a slow-speed handpiece and manually used to polish 14 specimens of each material. Two successive rubber points were used: the brown coarse points followed by the green fine points for 20 seconds each. This procedure was followed by the same two additional stages: pumice powder in water with a brush, and the final stage of zinc oxide and ethanol applied with a rubber cup (as described above).

## Profilometric Measurements

Profilometric measurements were performed using a stylus profilometer (Suretest Model 401, Mitutoyo Corp, Tokyo, Japan). This instrument was capable of measuring surface deviations to a resolution of 0.01  $\mu\text{m}$ . Three parallel scans were made across the central area of each sample. The samples treated with a finishing bur, either in a high-speed handpiece or in a low-speed handpiece, were tested in two directions: parallel and perpendicular to the rotation of the bur. All other specimens were tested in one direction: perpendicular to the rotation of the finishing instruments.  $R_a$  values (defined as the mean of the peak-to-valley displacement measured across the entire scan in  $\mu\text{m}$ ) and  $R_{\text{max}}$  values (defined as the maximum peak-to-valley displacement along the entire scan in  $\mu\text{m}$ ) were calculated for each scan by the instrument. Statistical analysis of both  $R_a$  and  $R_{\text{max}}$  was done with Repeated Measures Analysis of Variance, and comparisons were made with the Scheffé Rank Test. A sample from each polishing

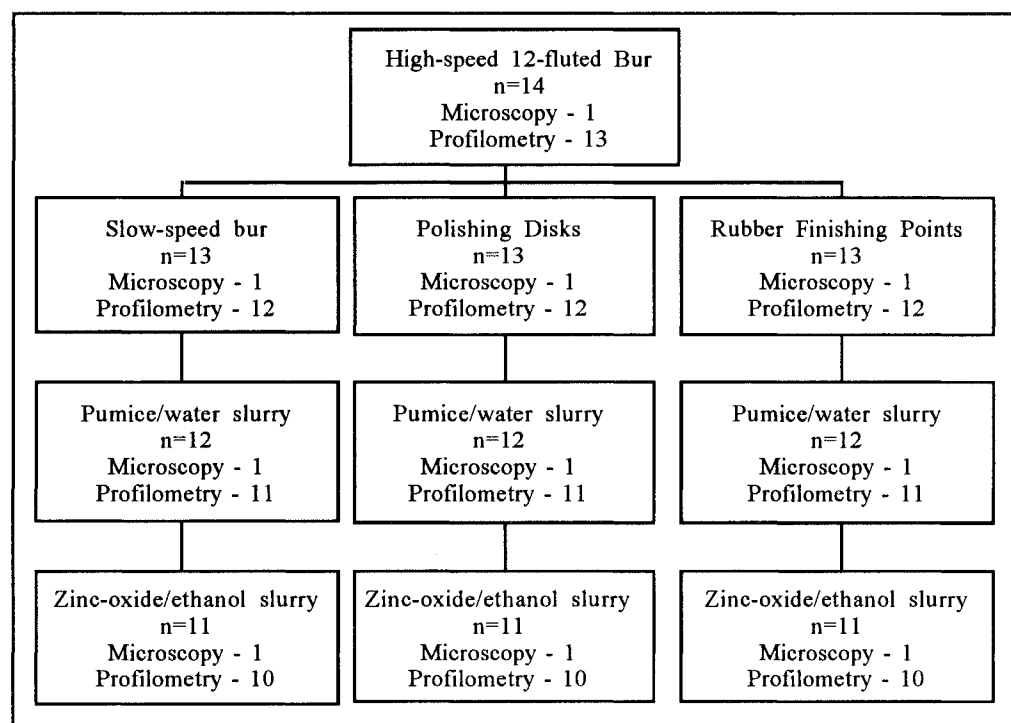


Figure 1. Order for the finishing methods and number of samples for each of the three metal surfaces

method was kept for further microscopic observation and photographed under reflected light at a magnification of X50. A flow chart of the order of finishing methods and number of samples for each material is illustrated in Figure 1.

## RESULTS

### Profilometric Measurements

Figure 2 shows the Ra values, and Figure 3 shows the Rmax values obtained after the different polishing procedures. Overall, amalgam had lower surface roughness values with most of the different treatment methods, and consolidated silver had the highest surface roughness values. The surface roughness (both Rmax and Ra) of the consolidated silver treated with the 12-fluted bur mounted on a high-speed handpiece was significantly greater than both alloys when measured in a direction perpendicular to the rotation of the bur (HS/Perp,  $P \leq 0.05$ ). The results obtained when measured parallel to the rotation of the bur (HS/Paral) were not significantly different for any of the three materials for either Ra or Rmax. The consolidated silver polished with a bur mounted on a low-speed handpiece had significantly higher Ra values than both alloys ( $P < 0.0167$ ) when measured in the perpendicular direction, and the consolidated silver Ra value was higher than gallium alloy when

measured in the parallel direction.

The three metals polished with disks (D) resulted in the lowest surface roughness values overall. The surfaces polished with rubber points (R) had slightly higher roughness values than disks, followed in roughness by surfaces polished with the burs (B). There was no statistical difference between the surface roughness of the three metals with either disk or rubber point polishing.

Additional polishing with pumice after the slow-speed bur (B/P compared to B/Perp) resulted in significantly smoother Ra values ( $P < 0.0001$ ) in all three metals. The addition of pumice polishing after the treatment with the disks (D vs D/P), re-

sulted in rougher Ra values ( $P < 0.05$ ) for the two amalgams. There were no significant differences in Rmax values. The addition of pumice polishing after treatment with rubber points (R vs R/P) resulted in smoother Ra values ( $P < 0.01$ ) for all three metals.

Further polishing with zinc oxide did not result in significant improvement in the smoothness (Ra) of samples previously polished by any of the three previously pumice-treated surfaces (B/P/Z vs B/P, D/P/Z vs D/P, R/P/Z vs R/P). There was no significant difference between the smoothness (Ra) of the three metals when polished by disks alone or rubber points alone. Further polishing with pumice did not result in any differences between the metals, and the final polish with zinc oxide did not result in any differences between the metals. The surfaces polished with the disks (D) resulted in similar Ra values to the rubber points and pumice (R/P) for all three metals.

### Microscopic Examination

Under visible microscopy, the serrations resulting from the 12-fluted bur were visible, though slightly more prominent in the silver specimen when compared to the amalgam and gallium alloy. The consolidated silver samples that were treated with the slow-speed bur also exhibited more prominent serrations when compared to samples of gallium alloy and amalgam. The visible scratches that resulted from the disks were also prominent in all three metal surfaces, even though these surfaces provided the smoothest



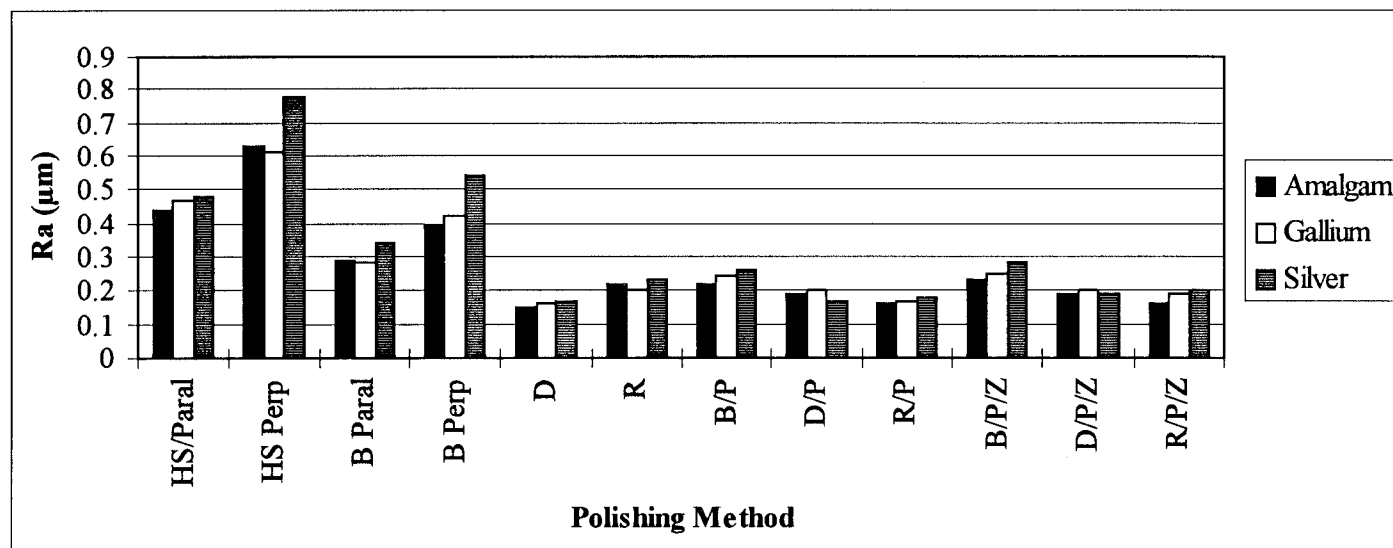


Figure 2. Mean  $R_a$  values (average peak-to-valley measurement across the scan) in  $\mu\text{m}$  achieved with the different polishing methods on the metal surfaces. The standard deviation for these measurements, an estimate of the standard uncertainty, did not exceed 8% of the mean. HS/Paral = high-speed 12-fluted bur measured parallel to the rotation; HS/Perp = high-speed 12-fluted bur measured perpendicular to the rotation; B/Paral and B/Perp = slow-speed bur measured parallel and perpendicular to the rotation; D = composite polishing disks; R = rubber polishing points; B/P = slow-speed bur + pumice/water slurry; D/P = polishing disks + pumice/water slurry; R/P = rubber points + pumice/water slurry; B/P/Z = slow-speed bur + pumice/water + a zinc-oxide/ethanol slurry; D/P/Z = disks + pumice/water + zinc-oxide/ethanol slurry; R/P/Z = rubber points + pumice/water + a zinc-oxide/ethanol slurry.

profilometric tracings. The treatment with the rubber points resulted in a notable reduction in surface roughness, but some residual bur serrations were still evident in all three metallic samples. The swirled scratches from the pumice powder were notable on

all samples, regardless of the type of metal surface, and the smoothness appeared comparable among the three metals. There was a marked change in the surface gloss of several samples that had received the additional zinc-oxide polish. This improvement in the

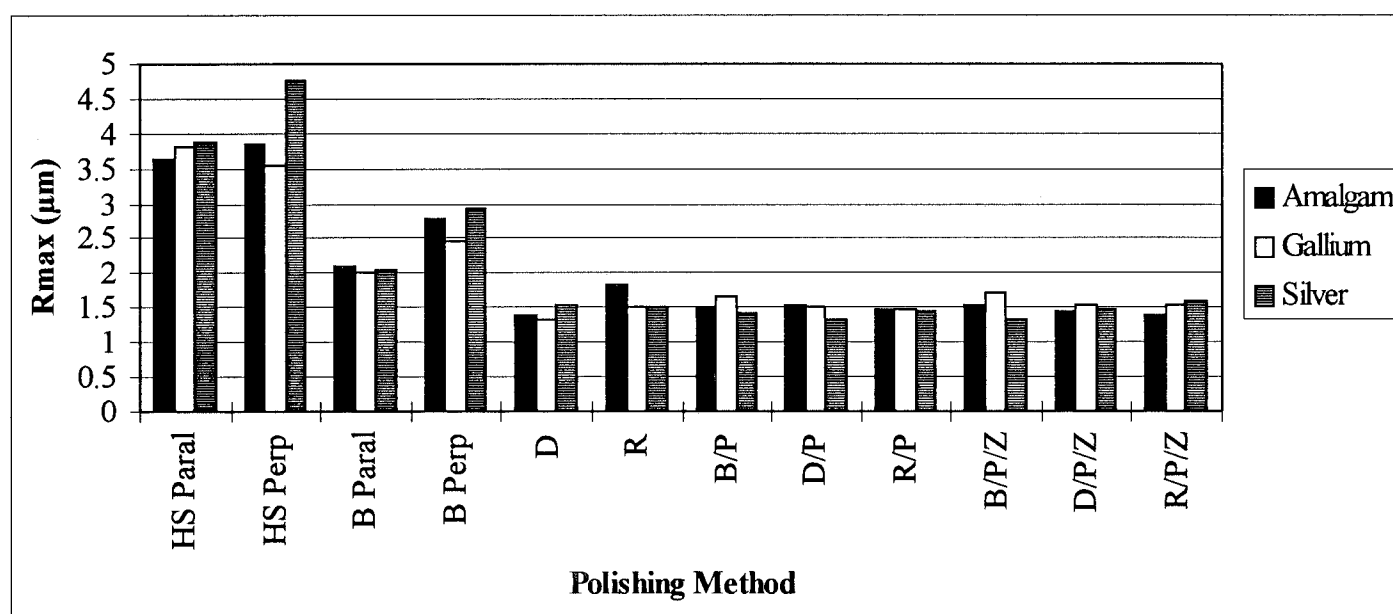


Figure 3. Mean  $R_{max}$  values (maximum peak-to-valley measurement across the scan) in  $\mu\text{m}$  achieved with the different polishing methods on the metal surfaces. The standard deviation for these measurements, an estimate of the standard uncertainty, did not exceed 10% of the mean. The identifications used for each of the finishing techniques are the same as those used in Figure 2.

surface appearance did not, however, correspond with smoother profilometric tracings.

## DISCUSSION

Consolidated silver has been recently studied as an alternative, mercury-free metallic restorative material. The physical properties of the consolidated silver have been extensively studied (Dariel & others, 1995). The delivery methods, instrumentation, and clinical properties are still being evaluated and will be critical to make the material feasible for clinical use. It is known, however, that rotary finishing, rather than carving, will be necessary to contour this cold-welded metal. Overall, it was demonstrated that for most finishing systems, the differences in surface smoothness among the three metals was small or not significant. Since most restorations require the formation of complex surface profiles, rotary finishing burs will be a necessity. Following this with a final polish using rubber points and pumice flour might be more feasible for clinical use, because polishing by disks is generally limited to fairly flat, accessible surfaces, such as class 5 restorations. Class 1 and class 2 restorations require the finishing of curved surfaces in order to adapt to the anatomical form of the teeth and may be more readily accomplished with the rubber points and pumice.

Additional polishing with the zinc-oxide powder and ethanol did not measurably improve the surface smoothness, even though it appeared visually that the gloss of the surfaces had improved. It may be that the particle size difference between the pumice flour and zinc oxide was small enough that the change in smoothness resulting from the zinc oxide was beyond the resolution (0.01 gm) of the profilometer.

The overall trend in relative smoothness among the metals was consistent for most of the finishing stages. Generally, amalgam exhibited the best polishability, followed by the gallium alloy and silver alloy. This order may correspond to the relative surface hardness of the three materials. The high degree of toughness and malleability found in silver makes it easy to produce deep scratches with coarse finishing systems that may not be completely removed by the subsequent polishing steps used in this study.

From these results, a finishing procedure for consolidated silver can be suggested: After condensation and burnishing, rotary finishing burs mounted on a high-speed handpiece can be used for shaping the occlusal surfaces and for occlusal adjustment. Disks and finishing burs can also be used to establish approximal contours where needed. This can be followed by finishing with rubber points mounted on a low-speed handpiece, and a final polish with a pumice-flour slurry applied with a rotary brush or cup.

## CONCLUSIONS

Polishing methods used for conventional amalgam provide similar smoothness for gallium alloy and consolidated silver. Contouring of consolidated silver restorations with 12-fluted burs followed by polishing with rubber points or disks and a pumice-flour slurry will provide a procedure that can establish the restoration contour and provide a surface finish comparable to that of similarly polished amalgam. Additional polishing with zinc oxide may provide a more glossy appearance, but will not measurably improve surface smoothness.

## Disclaimer

Certain commercial materials and equipment are identified in this paper to specify the experimental procedure. In no instance does such identification imply recommendation or endorsement by the National Institute of Standards and Technology or the ADA Health Foundation, or that the material or equipment identified is necessarily the best available for the purpose.

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# LITERATURE REVIEWS

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## Successful Photocuring: Don't Restore without It

W H TATE • K H PORTER • R O DOSCH

### Clinical Relevance

Photocuring is becoming an integral part of restorative dentistry. Proper curing-light function must be understood, monitored, and maintained to assure successful photocuring of restorative materials within a prescribed time.

### INTRODUCTION

Dentistry has an ever-expanding variety of restorative materials that require curing (polymerization) by photoactivation. Advances in composite resins, composite and alloy bonding systems, compomers, and hybrid ionomers (resin-modified glass ionomers) (McLean, Nicholson & Wilson, 1994) are making the light-curing unit as indispensable as the amalgamator. Various parameters of successful photocuring such as light intensity and time of curing influence the degree of material polymerization. The curing light must generate a suitable output intensity at the correct electromagnetic wavelength for an adequate duration of time. Studies have shown that the light

intensities produced by many curing lights in private practices are inadequate and cannot ensure maximum polymerization (Friedman, 1989; Barghi, Berry & Hatton, 1994; Caughman, Rueggeberg & Curtis, 1995; Miyazaki & others, 1998).

### PHOTOCURING DENTAL MATERIALS

Dental materials have progressed from requiring energy from the invisible ultraviolet spectrum (wavelengths from 10 - 380 nm) to the visible spectrum (wavelengths from 400 to 700 nm) for photocuring (Lee & others, 1993; Poulos & Styner, 1997). Specifically, the light at the blue end of the visible spectrum, near the blue-green border, polymerizes light-cured dental restorative materials. An optical filter, installed within curing lights between the bulb and the fiberoptic light-guide, allows only the lightwaves in the 400-520 nanometer (nm) range to pass through (Barghi & others, 1994). Photocuring is initiated by electromagnetic wavelengths between 400 and 520 nm. The green, yellow, and red range (520-720 nm) will not polymerize restorative materials (Caughman & others, 1995). The absorption spectrum of the most common photoinitiator in visible-light-activated composite resin (camphoroquinone) lies in the 450-500 nm wavelength range, with peak absorption at

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470 nm (Lee & others, 1993; Pires & others, 1993).

Curing-light intensity is termed "power density" and is measured in milliwatts per square centimeters ( $\text{mW}/\text{cm}^2$ ). The intensity of the light affects the physical properties of the restorative material and may also affect tooth-restoration bond strengths (Strassler, 1992; Pires & others, 1993; Lee & Greener, 1994; Unterbrink & Muessner, 1995; Shortall & Harrington, 1996a; Feilzer & others, 1995; Bouschlicher, Vargas & Boyer, 1997). Factors influencing the transmission of light throughout restorative materials include the thickness of the restorative material, the presence and size of filler particles (0.01 to 1 micron particles reduce light penetration due to light scattering), the shade of the restorative material (darker shades adversely affect transmitted light), cavity design, and the distance of the light tip to the restoration's surface (Barghi & others, 1994; Kawaguchi, Fukushima & Miyazaki, 1994; Caughman & others, 1995; Shortall & Harrington, 1996a,b). Polymerization of the restorative material will generally decrease from the surface of the restorative material inwardly (Strassler, 1992; Rueggeberg & others, 1993). As a result, apparent surface hardness is not an adequate indicator of complete material cure (Poulos & Styner, 1997; Shortall & Harrington, 1996b). Care must be taken to avoid the inadequate internal cure of a restorative material that appears to be cured on the external surface. Polymerization of the outer surface may be near maximum even with a light performing at only 30% effectiveness (Barghi & others, 1994).

It is generally accepted that an intensity reading of  $300 \text{ mW}/\text{cm}^2$  or greater in the proper wavelength range is needed for complete polymerization of materials up to 2 mm in depth (Bayne & Taylor, 1995), although several authors suggest a minimum intensity of  $400 \text{ mW}/\text{cm}^2$  for routine polymerization (Rueggeberg, Caughman & Curtis, 1994; Manga,

Charlton & Wakefield, 1995). Some investigators caution that high-intensity light may demonstrate significant disadvantages due to increased shrinkage stress (Unterbrink & Muessner, 1995; Feilzer & others, 1995; Venhoven, DeGee & Davidson, 1996); however, others have not observed significant differences between the maximum polymerization shrinkage forces generated by a high-intensity light versus a medium-intensity light (Bouschlicher & others, 1997). Still, rapidly developing polymerization contraction forces may build more quickly than the simultaneously forming dentin bond strength (Bouschlicher & others, 1997). Such an occurrence could lead to a partial or complete fracture of the bond at the tooth-restoration interface (Feilzer & others, 1995; Venhoven & others, 1996). At least one dental manufacturer offers a light-curing system with two-step polymerization, a low-intensity light phase followed by a high-intensity light phase (ESPE America, Norristown, PA 19404). Future studies and ongoing materials development should, at some point, clarify and allay at least some of these concerns.

Generally, an exposure time of 60 seconds will provide optimal polymerization if all parameters are adequate (Caughman, Rueggeberg & Curtis, 1995; Fowler, Swartz & Moore, 1994; Rueggeberg, Caughman & Curtis, 1994); however, undercuring has been observed even with an optimal technique and with a properly functioning light unit (Pires & others, 1993). Overall, when duration of time is considered, overcuring is not possible, only undercuring. Increasing the curing times for lights not operating at capacity sometimes will provide an adequate cure, although some lights operating at a very low efficiency may be inadequate even with increased curing time (Barghi & others, 1994; Strassler, 1992).

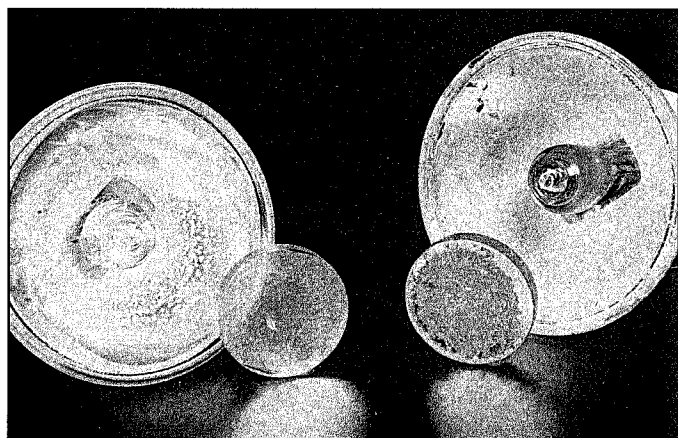


Figure 1. Damaged bulbs and filters

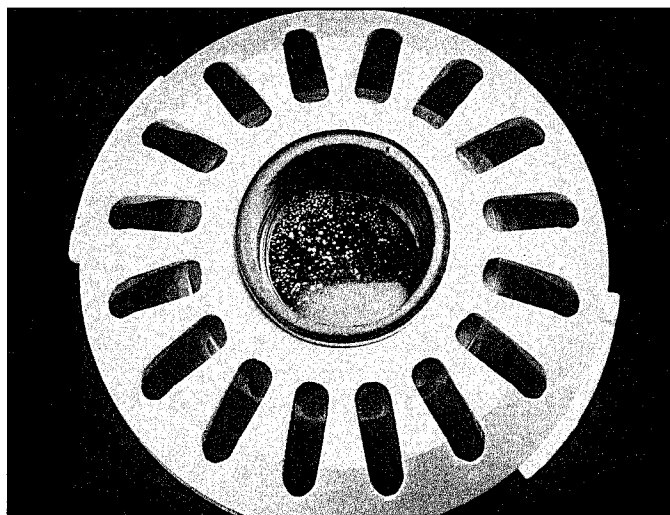


Figure 2. With light-guide removed, the external surface of an optical filter that is beginning to show degradation (pitting)

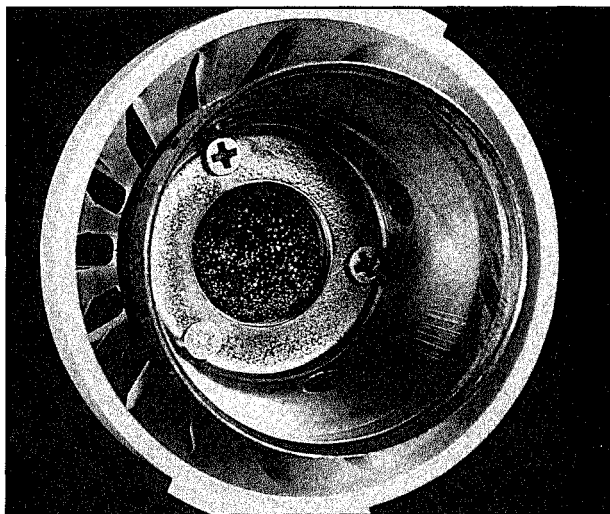


Figure 3. Internal surface of the optical filter pictured in Figure 2, showing pitting

### CURING-LIGHT PERFORMANCE

Curing-light performance will decrease over time because of various physical conditions. These conditions include bulb frosting or darkening, bulb degradation, optical-filter deterioration (pitting, cracking, and blistering), light-reflector degradation, breakage of the fiber-optic bundles, light-guide fracture, composite build-up on the light-guide tip, method of light-guide sterilization, line-voltage inconsistencies, and procedural factors such as direction, location, and size of the curing tip, variations in curing-light design (wand vs gun), movement of the curing tip, and the use of improper curing times (Strassler, 1992; Barghi & others, 1994; Fowler & others, 1994; Caughman & others, 1995; Shortall & Harrington, 1996b; Rueggeberg, Caughman & Comer, 1996; Poulos & Styner, 1997) (Figures 1-3). These detrimental conditions or combinations thereof influence the character and/or intensity of the transmitted light, thereby reducing the possibility of maximum material polymerization.

Frosting is caused by vaporization and condensation of the cement that holds the bulb and base together or by crystallization of the quartz portion of the bulb (Poulos & Styner, 1997). When this occurs, light intensity can be decreased by 30 to 40% (Strassler, 1992; Poulos & Styner, 1997).

Degradation of the bulb and reflector and delamination of the filter coating are mainly attributed to curing-unit overheating (Friedman, 1989; Caughman & others, 1995). Overheating can be minimized if the unit's fan is in good condition and is allowed to run its entire cycle without being interrupted. Periodic vacuuming of the fan with a suction tip can help eliminate

dust and maintain efficiency (Poulos & Styner, 1997). Initiation of a maintenance schedule for bulb and filter replacement after 2 years or 400 restorations, depending on the unit's monitored performance record, has also been suggested (Poulos & Styner, 1997). Bulbs and filter replacement kits can often be obtained from the manufacturer.

Trauma from normal use or from disinfection and sterilization procedures can cause fractures within the light-guide or break the fiber-optic bundle (Dugan & Hartleb, 1989; Rueggeberg & others, 1996; Nelson & others, 1997; Poulos & Styner, 1997). One glutaraldehyde-based immersion medium was shown to cause an irreversible structural breakdown of the surfaces of the glass rods in fiber-optic light-guides, consequently affecting light transmission (Dugan & Hartleb, 1989; Nelson & others, 1997). Other glutaraldehyde-based solutions did not significantly affect light-intensity transmission (Nelson & others, 1997). Although the glutaraldehyde-based solution that was found to be damaging has since been discontinued by the manufacturer, other groups of cold disinfection and sterilization solutions may pose similar problems. Water vaporized during autoclaving can deposit a mineralized residue, or boiler scale, on the light-curing tip. This scale build-up significantly decreases the ability of the light-curing guide to transmit light. In one study, after only three autoclave cycles, the intensity of transmitted light was reduced by 50% (Rueggeberg & others, 1996). However, the decreased transmitted intensity was returned to pretreatment values by completely cleaning the light tip using a commercial polishing kit (Demetron/Kerr Corporation, Danbury, CT 06810). Use of this polishing kit was also shown to greatly reduce the rate of boiler scale build-up on curing tips during subsequent autoclaving exposures (Rueggeberg & others, 1996). To eliminate the need for sterilization procedures and eliminate poststerilization performance concerns, single-use, disposable, presterilized curing probes are available for use with some curing units (Dentsply/Caulk, Milford, DE 19963).

Curing-light performance is also affected by other light-guide infection-control methods such as cellophane wrap (Chong & others, 1998; Warren, Rice & Powers, 1998). Any barrier-induced decrease in intensity may cause a light not operating at full capacity to fall below the recommended intensity and affect polymerization.

### MONITORING CURING-LIGHT PERFORMANCE

Consistent, accurate curing-light performance is essential and should be monitored routinely (Figure 4). Simply having a bright curing light does not ensure effective material polymerization. The curing

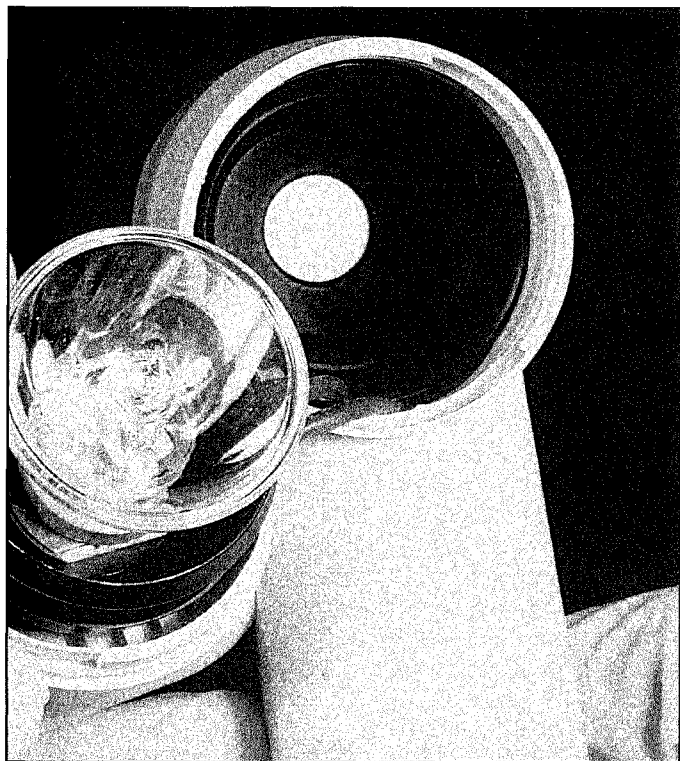


Figure 4. A light-curing unit with an intact bulb and undamaged optical filter (mirrored, gold internal surface)

radiometer is a useful instrument for obtaining instant measurements of power density ( $\text{mW}/\text{cm}^2$ ) from visible-light-curing units (Figure 5). Radiometers are designed to measure light intensity within a narrow wavelength range, but do not provide intensity versus wavelength (spectral distribution) information directly. However, it has been shown that measurements from a radiometer and a scanning monochromator used for spectrum analysis did correlate reasonably well, especially in the 450-500 nm range (Lee & others, 1993). A significant linear correlation between light-intensity readings from a radiometer and the degree of conversion of a light-cured resin has also been observed (Peutzfeldt, 1994). Thus, as a result of these and other studies, the curing radiometer has been shown to provide an effective means of quickly assessing the curing efficiency of light-curing units (Lee & others, 1993; Fowler & others, 1994; Shortall, Harrington & Wilson, 1995; Manga & others, 1995). Baseline intensity values should be established when the unit is new or when bulbs or components are replaced, since light-intensity varies from bulb to bulb and any new components may affect light transmission. Differences in light-intensity output exist between curing units made by different manufacturers and between units made by the same manufacturer



Figure 5. Radiometers, hardness disks, and split resin molds used in the monitoring of light-curing unit output

(Hansen & Asmussen, 1993; Manga & others, 1995). Routine monitoring throughout the life of the curing unit should be recorded and compared to the established baseline intensity (Strassler, 1992). Companies are now offering curing lights with built-in radiometers, which simplifies the monitoring of light intensity.

Two additional ways to measure the efficacy of curing lights is by the use of hardness disks (Demetron/Kerr Corp) (Strassler, 1992; Barghi & others, 1994) and the use of resin split molds (Ivoclar-Vivadent, Amherst, NY 14228) (Dunne, Davies & Miller, 1996) (Figure 5). Hardness disks have a thickness of 3 mm with a Barcol hardness of 75 and three 5 mm-in-diameter apertures. For each test one aperture is filled with resin and cured from the top side of the disk, following the manufacturer's guidelines. The surface hardness of the polymerized resin should match the surface hardness of the disk when tested with a carver on the top surface and on the opposite surface from the side exposed to the curing light (Barghi & others, 1994; Strassler, 1992). Hardness disks provide a tactile test, albeit subjective (Fowler & others, 1994), for the evaluation and monitoring of curing-light efficiency and intensity output. A resin split mold is available for depth of cure testing. The device contains two wells, one of 3 mm and one of 9 mm. Composite is placed into the wells and light cured. The mold can then be separated to assess depth of cure. Although the depth of cure using this resin mold has been shown to be deeper than in a natural tooth (Dunne & others, 1996), the device can give the practitioner an indication of curing effectiveness for routine curing-unit

monitoring (Shortall & Harrington, 1996b). For increased reliability of curing-unit monitoring, some suggest calibration of the radiometer and curing unit in regard to each other using depth-of-cure tests (Hansen & Asmussen, 1993). By correlating the curing efficacy of the curing unit with the corresponding radiometer reading, a reference for future monitoring and polymerization understanding and assessment is established.

## CONCLUSION

Light-curing units must be monitored on a regular basis even if the light output and the cured surface of the restorative material appear to be satisfactory. A curing radiometer, hardness disk, split resin mold, or preferably some combination of the three can be used to assess the adequacy of curing-unit performance. Physical examination and subsequent repair or replacement of damaged light-curing unit components should be included in the routine monitoring of curing lights. Routine monitoring and maintenance of dental light-curing units assure maximum restorative material polymerization and, therefore, successful photocuring of restorative materials within a prescribed time.

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# Glass Ionomers: A Review of Their Current Status

G J MOUNT

## INTRODUCTION

More than 20 years of clinical experience with glass ionomers has shown them to be versatile materials that are simple to handle and relatively tolerant of variations in clinical techniques (Mount, 1994). There is sufficient evidence in the literature to suggest that, properly handled, they have an excellent record for longevity (Mount, 1997; Matis, Cochran & Carlson, 1996). Like all restorative materials they have their limitations, and a clear understanding of their handling characteristics is essential for success. As a water-based cement with an acid/base setting reaction, the maintenance of the water balance is important but not difficult to achieve (Wilson & Nicholson, 1993). It is essential in the development of proper translucency when the material is to be used for an aesthetic restoration and is always necessary if full physical properties are to be developed and maintained.

Glass ionomers were designed in the first place as a replacement for silicate cements. In this they have been successful because they have many of the same properties along with some additional advantages. The main problem with silicates was that the profession failed to understand the essential requirements for success with them, and it seems it is still being slow in coming to terms with glass ionomers. Fluoride release and translucency of silicates have been repeated, but there is one significant advantage now apparent. That is the ion-exchange adhesion with tooth structure, which is unique in dentistry and of great value. It has been recognized for many years that microleakage between the restoration and the cavity wall is probably dentistry's greatest hazard, and this is prevented by the ion-exchange adhesion of glass ionomers (Mount, 1991; Ngo, Mount & Peters, 1997; Mount & Hume, 1998). The following discussion will cover the advantages, the currently accepted uses of glass ionomers, and their limitations.

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## IDENTIFICATION OF GLASS IONOMERS

First it is necessary to overcome some of the problems of identification that have arisen in the market place as a result of confusing advertising. Glass ionomers consist of an aluminosilicate glass and a polyalkenoic acid, which set through an acid/base

reaction between the filler and the resin. It can, therefore, be regarded in some ways as a type of composite resin where, unlike the standard resin composite, the filler takes part in the setting reaction. Based in part upon the powder-to-liquid ratio and in part on variations in the manufacturing process, the various types of material in this category, and their clinical applications, can be identified through the following classification (Mount, 1994):

Type I—luting cement with a fine powder particle size using a low powder content;

Type II.1—restorative aesthetic cement with high powder content and therefore higher physical properties. Acceptable translucency means that this group can be used in situations where aesthetics is of importance;

Type II.2—restorative reinforced cement with high powder content and higher strength. To be used where aesthetics is of no concern but strength is important;

Type III—lining or base cement. The lining cement is mixed with a low powder content so that it will flow readily but has lower physical properties. An increase in powder content will increase the physical properties and convert it into a base or dentin substitute.

### Resin-modified Glass Ionomers

It should be noted that both the Type II and Type III categories can be modified by the inclusion of small quantities of a resin such as HEMA, which, along with activators, will make them susceptible to light activation. However, the light-activation component must not overwhelm the acid/base reaction, because this is an essential part of the fluoride-release mechanism as well as the ion-exchange adhesion, and these are the hallmarks of a glass ionomer (Sidhu & Watson, 1995; Mitra, 1994). With the light-activated materials the fluoride release has been shown to be essentially the same as for the autocure materials (Forsten, 1994b). Also, the ion-exchange adhesion system is still available, and a resin bonding system must not be used (Desai & Tyas, 1996; McLean, 1996).

The amount of added resin varies between manufacturers but is in the vicinity of 5% in the finished set cement for a restorative material. It will be higher for a lining cement, which is mixed with a lower powder content. The advantages include immediate stabilization of the water balance following light initiation as well as enhanced translucency in the ultimate restoration. The main disadvantage appears to be the need for incremental build-up for a restoration deeper than 3-4 mm, because light penetration is limited to that depth. However, there is a safety factor inasmuch as the acid/base reaction

will continue, even in the absence of light activation. In addition the presence of a redox reaction in the resin component ensures complete setting of the entire restoration under all circumstances.

It must be noted that there appear to be some minor contraindications to the use of the resin-modified glass ionomers. The approximately 5% of resin that is normally added is HEMA, along with small quantities of a photoinitiator (camphorquinone). HEMA is strongly hydrophilic, and this means that these materials tend to perform like a mild hydrogel with rapid water uptake over the first 5-7 days after placement, leading to a small amount of expansion of the restoration and possible stain uptake in the short term (Nicholson, Anstice & McLean, 1992). Subsequently the resistance to wear appears to be lower than that shown by the autocure materials. There have also been reports of modification of color, although the clinical significance of these factors does not appear to be of great concern (Davis, Friedl & Powers, 1994).

### Compomers

Confusion has arisen in the market because of the introduction of the so-called compomers, which are essentially a resin composite in which the filler is a glass similar in many ways to the ionomer glass. There is also a variable quantity of a dehydrated polyalkenoic acid incorporated with the filler, but this does not become available to react with the glass until such time as there has been some water uptake into the restoration. The initial setting reaction is through a light-activation system identical with the standard resin composites. Subsequently there will be a limited degree of a glass-ionomer type acid/base reaction, which will release small quantities of fluoride. However, the adhesion system has to be based on the resin to dentin methods, because an ion-exchange adhesion cannot arise at any stage. Some manufacturers have made claims in advertising that suggest a close relationship between glass ionomers and compomers, but this is clearly not so. McLean, Nicholson, and Wilson (1994) set out a classification that defined the various materials and placed each into a clear perspective in an attempt to avoid confusion. However, it is still necessary for the profession to make sure to use the correct material in its proper place.

### ION-EXCHANGE ADHESION WITH TOOTH STRUCTURE

Providing the cavity surface is cleaned of debris and smear layer through proper conditioning prior to placement of a glass-ionomer material, there will be an immediate ion exchange between the cement and

the enamel or dentin (Mount, 1991; Ngo & others, 1997). The probable mechanism of adhesion is based upon both diffusion and adsorption phenomena. The polyalkenoic acid of the glass ionomer will penetrate the tooth structure, releasing phosphate ions, each of which will take with it a calcium ion from the tooth surface to maintain electrical neutrality. These ions will combine with the surface layer of cement and form an intermediate layer of a new material, which is firmly attached to the tooth surface (Geiger & Weiner, 1993). This has been described as a "diffusion based adhesion" (Akinmade & Nicholson, 1993). There is also a degree of adhesion available to the collagen of dentin through either hydrogen bonding or metallic ion bridging between the carboxyl groups on the polyacid and the collagen molecules (Akinmade, 1994). This means that the dentin does not have to be fully mineralized to achieve a chemical union with a glass ionomer.

The question of conditioning the tooth surface in preparation for adhesion has been widely debated. Originally 50% citric acid was recommended, but this was abandoned in favor of 10% polyacrylic acid (Wilson & McLean, 1989), which has been shown to effectively clean the surface within 10 seconds. The advantage of this particular acid is that it is part of the glass-ionomer system, so any remaining residue will not interfere with the setting reaction of the cement. In addition it will alter the surface energy of the tooth, thus encouraging adaptation of the cement to the cavity walls. Stronger acids should not be used for conditioning because they may unduly demineralize the tooth and reduce the efficiency of the ion-exchange adhesion.

In the presence of this type of adhesion, failure, leading to loss of a restoration, will be cohesive in the cement, rather than adhesive between the two materials, so that a thin layer of ion-enriched material will remain firmly attached to the tooth. Thus, even after failure, the dentin tubules will still be sealed with an effective barrier that will continue to prevent microleakage.

This ion-exchange adhesion system is unique in dentistry and is of considerable significance. It means that the stronger the material the greater the adhesion, and this is shown clearly in bonding studies (McLean, 1996; Peutzfeldt, 1996). There has been considerable confusion in reports of bond strength, because the relatively slow maturation of glass ionomers is often not recognized, and they are tested too soon after placement. In addition the type of failure is often not identified. With a properly placed glass ionomer it will be cohesive in the cement, and careful observation will show that there is a thin layer of material left attached to the tooth that will leave the surface sealed (Øilo, 1993). Therefore, a restoration that is likely to be placed under heavy occlusal

load should have the highest possible powder content to ensure optimum physical properties. Cervical and occlusal restorations will last well, providing they are mixed at the optimum ratio and are well supported by remaining tooth structure (Matis & others, 1996).

## FLUORIDE RELEASE

One of the important properties that glass ionomers share with silicate is the release of fluoride ions throughout the life of the restoration (Forsten, 1994a). Silicate cement was always noted for the fact that, although it was notorious for washing out, there was rarely any recurrent caries in the cavity for quite some time after its loss. Glass ionomer also has a reputation for providing resistance to further demineralization to surrounding and adjacent tooth structure (Segura, Donly & Stratmann, 1997).

Fluoride is incorporated in the glass during manufacture to act as an oxide scavenger and to modify the fusion temperature of the glass melt (Wilson & McLean, 1989). It will also have an indirect bearing on the reactivity of the ultimate glass powder. The quantity can be varied and will have a bearing on translucency of the set cement, the working characteristics, and the ultimate physical properties.

Immediately upon mixing the glass powder with a polyalkenoic acid, the fluoride ions will be released by the initial attack of the acid on the surface of the glass particles. The fluoride ions will take no further part in the setting reaction but will remain within the matrix that is formed by the development of calcium and aluminum polyalkenoate chains. The acid/base setting reaction will continue for some time, and considerable change will occur over the first hour. Maturation will not be complete for some months but will have reached an advanced stage within the first week. Even after completion of maturation, the matrix remains porous to the extent that the smallest ions, such as the hydroxyl and the fluoride ions, can move freely through it. The glass particles are also porous to some degree, so the ions can move freely through the entire restoration. Understandably there will be a considerable release of fluoride ions from a newly set restoration within the first few days, and a relatively generous flow will continue for a month or more thereafter. The release will then stabilize and continue at a low level for many years (Forsten, 1994a).

It has also been shown that a subsequent professional application of a topical fluoride will result in a further uptake of fluoride ions into a restoration, and this will be followed by an increase in the rate of release for a short period. Thus a glass ionomer can be regarded as a fluoride reservoir within the oral environment (Forsten, 1993, 1994b),

and this is another property that is unique to these materials.

As a result, resistance to demineralization in immediately surrounding tooth structure has been reported, as well as modifications to plaque build-up on the surface of glass-ionomer restorations (Serra & Cury, 1992). It has been shown recently that the ion release is sufficient to buffer the acid level of plaque in the vicinity and thus reduce the potential for demineralization (Nicholson, Czarnecka & Limanowska-Shaw, 1998). This is not surprising in view of the fact that relatively low levels of fluoride have been shown to be a catalyst in the process of remineralization of the carious lesion (Silverstone, 1982). Also some bacterial species fail to thrive in the presence of fluoride, particularly *Streptococcus mutans*, which is commonly found in plaque (Meiers & Miller, 1997). Clinical observation confirms that the development of recurrent caries is unlikely in the presence of a glass-ionomer restoration (Tam, Chan & Yim, 1997). Obviously, complete inhibition should not be expected, and all restorative dentistry should be undertaken in conjunction with accepted normal preventive procedures.

### BIOCOMPATABILITY

None of the existing restorative materials is, in itself, irritating to the pulp, although some of the individual constituents, applied alone, may produce an adverse response. Glass ionomer is no exception to this, and it is well tolerated, even by healthy exposed pulp tissue (Cox & Suzuki, 1994; Hume, 1994). The main problem in restorative dentistry is microleakage between a restoration and the cavity wall. If bacteria and their toxins are able to penetrate down the interface, there will be a rapid inflammatory response in the pulp (Brännström, 1987). However, rapid recovery is also possible as long as the bacteria can be eliminated. This means that because of the ion-exchange adhesion, which will prevent microleakage, glass ionomers are of considerable value in isolation of an active carious lesion. Massler (1967) demonstrated the use of zinc oxide and eugenol paste in sealing a carious lesion and noted the subsequent rapid elimination of inflammation from the pulp. This system worked because eugenol is highly bactericidal, rather than through effective sealing of the margin. He noted recovery of the pulp within 3 weeks after sealing, and the same effect has been demonstrated with glass ionomer. With this material it works because of an effective seal rather than through any antibacterial action, but the result is the same.

Zinc oxide and eugenol will stabilize a lesion and allow the pulp to overcome the inflammation. There is then likely to be some deposition of secondary dentin on the roof of the pulp chamber, providing

protection against further bacterial insults. Clinical observation suggests that glass ionomer will produce a similar result in sedation of inflammation and, in addition, it appears to stimulate remineralization of the demineralized affected dentin as defined by Massler (Ngo, unpublished report). This is considered to be logical on the grounds that there may be a release of ions other than fluoride ions, such as calcium, strontium, and phosphate ions, from a glass ionomer, and these may be utilized in the remineralization process.

It has been shown in vivo that there is considerable remineralization of both enamel and affected dentin in the presence of a glass-ionomer temporary restoration (ten Cate & van Duinen, 1995). It is suggested, therefore, that the profession should re-examine the progress of the carious lesion. Massler defined the infected and affected zones and suggested that the affected should be retained, at least until the pulpal inflammation had subsided. It is now suggested that the affected dentin should be regarded as "pre-carious," rather than actively carious, and retained until such time as the body has had a reasonable opportunity to remineralize it as far as possible.

Remineralization is not difficult in the presence of more recent versions of glass-ionomer materials, particularly those which have improved physical properties. There are now available fast-setting autocure materials, with acceptable aesthetics, that are simple to place and are immediately resistant to water uptake as soon as they are set. It is sufficient to open the cavity wide enough to allow removal of infected dentin and the development of clean enamel and dentin walls around the periphery (Mount & Hume, 1998). Affected dentin should remain on the pulpal floor, and the cavity should be conditioned as usual. A glass-ionomer restoration can then be placed using a high powder content material for optimum physical properties. If this restoration is left in place for at least 3 months, it will be found, following removal, that there is a relatively sound, well-mineralized, floor remaining, and a final restoration can be designed, based on a minimal cavity design.

### CLINICAL PLACEMENT

Contrary to the common perception, these materials are relatively simple to place in a clinical situation and are certainly no more difficult than amalgam or resin composites (Mount, 1993; Naasan & Watson, 1998). They have been shown to be long lasting in relatively minor restorations and the material of choice for the modern-concept minimal preparation cavities (Wilson & McLean, 1989; Mount, 1994). The following factors should be noted for effective placement.

### Ease of Handling

Glass ionomer is a relatively simple material with a straightforward acid/base setting reaction that is well known in dentistry (Wilson & Nicholson, 1993). As with all two-part materials, proportioning of the two components is important for the development of the full potential of physical properties. Maintenance of correct mixing time and working time is also relevant to success (Mount, 1993).

The old methods of dispensing and mixing by hand have been abandoned in relation to most dental materials, and glass ionomers are no exception. Most of them are now available in a capsulated form, thus eliminating one of the variables that could lead to poor results. While most of the glass ionomers are also available in powder and liquid form, the capsulated version is strongly recommended because of the simplification of handling, the ease of placement, and the increased reliability for quality results. Most capsule systems also convert to a syringe, which means that the cement can be reliably placed to the depths of a cavity, particularly those minimal cavities discussed below.

Physical properties such as compressive and tensile strength are directly related to the powder content of the mixed cement, and the distribution of powder particle size can be varied. Also the cement is thixotropic in nature and flows well under pressure. A Type I luting cement will have a small particle size and a low powder content. Having applied optimum pressure to ensure proper adaptation of the prosthesis to the prepared cavity, the pressure can then be released, and the cement does not show a delayed elastic memory that, with other cements such as zinc phosphate cement, is likely to displace the restoration (Mount, 1993).

The main difference between a lining and a base (dentin substitute) is the proportion of powder to liquid. A low powder content mix can be placed as a lining under a conventional restorative material. It will flow readily and set rapidly, achieving satisfactory physical properties in a reasonably short period of time. However, such a mix should be not used for the lamination technique where a resin composite is to be placed over the glass ionomer. Under these circumstances it is necessary for the glass ionomer to be as strong as possible, to resist the setting shrinkage of the resin, so a high powder content mix is essential. It can now be regarded as a dentin substitute.

For all other restorative techniques where glass ionomer is indicated, such as fissure sealing, laminations, restorations both long term and temporary, it is necessary to use the strongest material available and therefore with the highest powder content recommended by the manufacturer. Recent reports on

longevity suggest that there is no problem, providing the material is mixed properly, the cavity is conditioned correctly, and the water balance is maintained through the early stages of setting (Mount, 1994).

### Development of Translucency

One of the earliest uses for glass-ionomer restorative materials was for the restoration of cervical erosion lesions where the ion-exchange adhesion meant that preparation of a retentive cavity was not required. The challenge was to develop a suitable translucency in the cement, and it was shown that maintenance of the water balance was an essential element for success (Mount, 1990, 1993). The first glass ionomers were not translucent at all, and it is suggested that the premature marketing of the material before it was fully developed may have had a negative effect on its future use. However, within a brief period the manufacturers produced products that were shown, on the laboratory bench, to have very satisfactory optical properties. The problem was to transfer these advantages to the oral environment.

Since glass ionomers are water based, water plays an essential part in hydrating-reaction products such as the metal polyalkenoate salts and silica gel. It is accepted that water is present in a "tightly bound" form, and this cannot be lost from the setting reaction (Wilson & McLean, 1989). However, in the early stages of setting, there is also a reasonable quantity of "loosely bound" water present, and this can be lost quite readily if the restoration is not sealed. During this early phase it is also possible for the restoration to take up available water from other sources, which will in turn wash out metal ions, particularly calcium, because the newly forming calcium polyalkenoate chains are highly soluble in water. The ultimate physical properties will be reduced and the translucency destroyed.

### Maintenance of the Water Balance

Maintenance of the water balance is therefore essential for the development of the full aesthetic properties of the Type II.1 restorative aesthetic materials and is not difficult to develop in the clinical environment. Following the initial set of the cement and removal of the matrix, the restoration should be covered immediately with a generous layer of a single-component, low-viscosity, light-activated resin bond (Earl, Mount & Hume, 1989). The restoration can be trimmed through the unset bond and further sealant added if required. Finally the bond can be light activated to provide a long-lasting waterproof seal. Making sure that there is no excess left along the gingival margin to form an overhang, the seal

will be effective for at least 24 hours. Contouring and polishing should be delayed until this time, although the best surface finish will be developed by the matrix itself and should be left undisturbed if possible. The end result will be the development of excellent translucency combined with acceptable physical properties (Mount, 1997). As the bond is an unfilled resin, it will wear off the surface quite rapidly and will not interfere unduly with the subsequent fluoride release.

The fast-setting materials listed in the other categories of the classification do not need to be sealed against water uptake, but this same sealant should be used under any circumstances in which water loss may be a problem. The glass in these cements has been chemically modified during manufacture to reduce the calcium content and thus limit the production of calcium polyalkenoate chains, which are highly water soluble. This will allow for a faster maturation of the setting reaction but will, in turn, reduce the translucency.

The light-initiated resin-modified glass ionomers are resistant to water uptake the moment they are light activated (Mount, 1990). However, it is a good idea to seal a newly placed restoration, as described above, simply to eliminate surface scratches and roughness following contouring.

### Minimal Cavity Design

In view of the above discussion concerning the potential for remineralization, it is now possible to review cavity designs, particularly for new lesions of limited extent. In the past, the approach to cavity design has been based upon surgical removal of the entire carious lesion as well as additional tooth structure that may be regarded as susceptible to further caries (Black, 1917). In the absence of adhesion it was then necessary to develop retentive designs within the cavity to ensure that the restoration was mechanically retained.

The situation has now changed because of the development of micromechanical adhesion to enamel using resin composite and chemical adhesion to both enamel and dentin using glass ionomer. There is no longer a need to develop retentive designs, and "extension for prevention" is a principle of the past. Cavity design for replacement dentistry, in which failed restorations are reconstructed, will be dictated by the existing cavity design and generally cannot be so conservative. But the design of a cavity to repair a new lesion can be very conservative, providing always that all normal preventive measures are undertaken at the same time to try to eliminate caries activity in each particular patient.

The micromechanical union between enamel and resin composite is very strong indeed and is limited

only by the strength of the enamel itself. Providing it is well mineralized and supported by sound dentin, it will provide a long-term union. However, because of the heterogeneous nature of dentin, and the inevitable presence of water within the dentin tubules, it has not been possible, up to now, to develop a sound long-term union between resin composite and dentin (McLean, 1996).

However, the ion-exchange chemical union between a water-based glass ionomer and tooth structure has been shown to be long term, with the main limitation being the relatively low fracture resistance of the cement itself (Mount, 1997). The best way at present to compensate for this perceived weakness is to utilize both materials to make a combined laminated restoration in which the glass ionomer provides the union with dentin and the resin composite reinforces it and provides the union with enamel (Mount, 1989). If the entire cavosurface margin is in sound, well-supported enamel, it is possible to seal the margin with a resin-to-enamel adhesion. On the other hand, if a high-strength Type II glass ionomer is used as the dentin substitute, it is possible to leave glass ionomer exposed to the oral environment, particularly at the gingival of the approximal box, and thus available for optimum fluoride release.

The additional advantages of glass ionomer include its potential for enhancing remineralization. This means that there is no longer a need to extend the cavity outline beyond contact areas with adjacent teeth, and neither is there a need to remove all demineralized enamel around a progressing lesion. It is only necessary to remove enamel that has been completely broken down, and surrounding enamel that is still intact but demineralized can be retained (Mount & Hume, 1998). Glass ionomer should be used to restore any defect in the enamel to the point where plaque can no longer accumulate and the surrounding demineralized but smooth enamel can be remineralized using both topical applications and the fluoride in the adjacent restoration.

The result of this approach to cavity design is the retention of considerable areas of natural tooth structure, which had to be sacrificed in the past just to achieve the required cavity outline. It is acknowledged that no restorative material is perfect, and longevity of a restoration is always a problem. But it is suggested that any system that extends the life span of natural tooth structure is desirable, even if it only adds one additional stage between the original restoration and the crown, which, at present, is almost inevitable.

### LIMITATIONS

It would seem from the above that the only serious limitation to the use of glass ionomer is its relatively



low fracture resistance. As with other dental restoratives, such as amalgam and resin composite, the set cement is essentially a filler supported by a matrix. The weakness appears to be in the matrix, which is prone to crack propagation under load, particularly in the presence of defects within the matrix. As a two-part material, which needs to be mixed prior to placement, it will inevitably contain a degree of porosity, and cracks tend to follow through these defects.

A second potential limitation lies in the fact that, until the material is mature, it is prone to dehydration and cracking. The fast-setting cements reach maturity within about two weeks of placement, but the Type II.1 autocure, aesthetic restorative cements remain susceptible for up to 6 months. Damage can be avoided simply by sealing a restoration with the low-viscosity resin seal if it is to be exposed to dehydration prior to maturation.

There are methods available to enhance the physical properties, and considerable research is being directed to this end. Already the materials available at this time show advances in physical properties, and further improvements are anticipated. Using a shear punch test as a method of assessment (Mount, Makinson & Peters, 1996), the more recent versions of the restorative materials are nearly three times as strong as the original glass ionomers of 20 years ago. On average, the early versions showed strengths in the range of 30-35 MPa. The next major change came with the advent of the resin-modified materials with strengths of up to 65 MPa. The most recent materials, the high-strength, autocure, fast-setting cements, recommended for long-term temporaries for adults or permanent restorations in deciduous teeth, have been recorded at 75-85 MPa. These figures must be compared with similar tests on resin composites where the microfills are in the range of 85-95 MPa and the hybrids reach up to 135 MPa. The differences are not great, and the alternative technique of lamination is always available if there is concern regarding the longevity of a restoration.

### THE FUTURE

Improvements are obviously desirable, providing they do not interfere with the ion-exchange adhesion, the release of fluoride and other ions, or the biocompatibility. It is apparent from the above discussion that the main avenue for research must continue to be in the field of enhanced physical properties. There must also be further investigation into the potential for a therapeutic effect upon the carious process. Although there have only been limited observations up to now, the results so far indicate a more positive therapeutic effect from glass ionomers than any of the other restorative materials

currently available. It is suggested that the following areas need to be researched.

### Reduction of Porosity

It is inevitable that there will be air incorporation in any material that is initially in two parts and requires mixing, whether by machine or by hand. Elimination of all porosity is probably impossible, and if it could be achieved the material would be too viscous to be handled easily in the oral environment. Reduction of the macroporosities could, however, be achieved by mixing at reduced atmospheric pressure or by using centrifugal force to "condense" the material. There are doubtless other methods that could be explored, and research is required.

### Modifications to the Glass

There are an infinite number of different constituents available with which to make glass, and very few combinations have so far been tested (Wilson & Nicholson, 1993). The fluoride content can be varied to have an effect upon the fusion temperature of the melt and thus on the heat history of the glass. This in turn will affect the reactivity of the powder as well as the translucency. There are many other variations possible in both the constituents and the relative proportions of the constituents. For example the substitution of calcium by either strontium or lanthanum will alter radiopacity. As strontium is very close to calcium on the periodic table, it can be expected to behave in a similar fashion and, in fact, it is possible to form a strontium apatite within normal tooth structure (Wilson, personal communication). Some manufacturers are already using a strontium rather than a calcium glass. A fast-setting cement with an early resistance to water uptake can be formed by removing excess calcium (strontium) ions from the surface of the powder particles during manufacture. The surface reactivity of the powder can be varied by chemical means and by introducing changes in the heat history of the melt. Recently, improvements in the physical properties have been shown to arise from control of the powder particle size and the size distribution. This technique has led to improvements in resin composites, and it may lead to further improvements in the glass ionomers.

### Modifications to the Liquid

Similarly there are many other polyalkenoic acids available, some of which may be of value in this system. At present the ones in common use include polyacrylic, itaconic, and polymaleic acids. A number of others have been investigated, including a

polyvinyl-polyacrylic acid (Ellis & Braybrook, 1990; Ellis & Wilson, 1992) but without success. It is known that increasing the molecular weight of the acid will increase the physical properties of the set cement, but it also increases the viscosity and therefore the problems of mixing and clinical handling. This problem has already been overcome to some degree by dehydrating the acid and incorporating it within the glass powder, then using water as the liquid for mixing. There may be other ways of incorporating the powder into the liquid, and variations on this theme should be investigated further.

### Disperse Phase Inclusions

The concept of incorporation of fine particles designed to prevent crack propagation is not new and has been tried already in dental materials. An early silicate cement had glass fibers included, and recently a similar approach was attempted with a glass ionomer. So far the end result has not been satisfactory, but the potential remains.

### Enhanced Therapeutic Effect

As already discussed above it would appear that glass ionomers will do more than just obturate a cavity. In fact, they appear to have a positive therapeutic effect upon the oral environment and the caries process. They are acknowledged to be a dynamic material in which there is a continuous maturation. The ion-exchange, diffusion-based, adhesion to tooth structure will prohibit microleakage and provide an ideal seal between the restoration and the tooth. The fluoride release has a positive effect upon remineralization and a negative effect upon plaque accumulation. The release of calcium and phosphate ions may enhance the remineralization potential as well as buffer the surrounding plaque. There also appears to be the potential for calcium and phosphate uptake as well, inasmuch as there is an improvement in surface hardness as the material matures in the mouth (Wilson & McLean, 1989). Further research is justified to see if these effects can be enhanced.

### CONCLUSIONS

It is apparent that glass ionomers can play a useful part in restorative dentistry. No one material is universal, and it is unlikely that such an ideal will ever be achieved. All our current materials have limitations, but each one, used to its full potential, has a place. There have been 20 years of clinical observation of glass ionomers, and their main advantages, as listed above, make them a valuable adjunct to restorative dentistry.

The ion-exchange adhesion is unique and particularly valuable in view of the fact that microleakage is such a problem with all other restorative materials. While the micromechanical attachment of resin composite is arguably the strongest union in dentistry, it is dependent upon the strength and condition of the enamel around the entire margin. An effective long-term union between resin composite and dentin has still to be perfected. As compomers require the use of a resin bonding agent, they must be placed within the resin composite category when discussing adhesion.

Other advantages such as biocompatibility and usefulness in minimal cavity design stem from these properties. It is important then, when selecting the appropriate material for a particular restoration, to consider the ultimate gain in maintenance and retention of natural tooth structure. It is clear that all new carious lesions should be approached with a minimal cavity design in mind. Glass ionomer will then be the material of choice because of the listed advantages, with the proviso that it be supported and reinforced with a stronger material, such as a resin composite or amalgam, only in a situation where the occlusal load is expected to be too great for glass ionomer to sustain. As long as there is sufficient remaining tooth structure available to surround and support the restoration, then glass ionomer will be the best material. Even when the cavity has extended beyond these minimal dimensions, it will still provide the best seal between the restoration and tooth structure.

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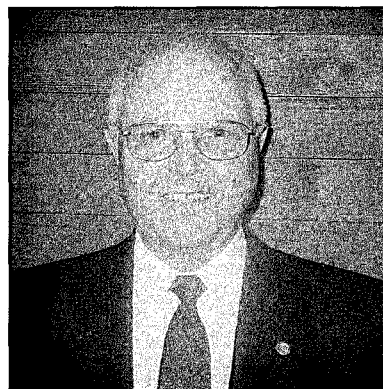
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## Clinician of the Year Award

The Clinician of the Year Award was donated by Vic Williams on behalf of the Ivoclar/Williams Company to recognize annually one of the Academy of Gold Foil Operators' members for his/her outstanding contribution to dentistry. I would like to thank Ivoclar for their continued support in making this award possible.

Dr Anthony Romano's presentation at the 1998 American Academy of Gold Foil Operators annual meeting gave us an excellent trip down memory lane. During his lecture, he brought to mind some of the great people who have formed the Academy and from whom we have had the opportunity to learn.

Dr Ralph Stenberg has become a quiet leader and a strong voice for clinical excellence. He has been a Husky all of his professional life, having gone to undergraduate and dental school at the University of Washington. There, he learned from the best and knew what excellence in restorative dentistry was all about by the time he graduated. He became an active member of several gold foil groups, serving as Past President and Secretary for both the Hampson Ferrier Gold Foil Study Club and the Associated Gold Foil Study Clubs of Oregon, Washington, and British Columbia. In his effort to achieve clinical excellence in restorative dentistry, he felt it was necessary to gain a greater understanding of occlusion. This resulted in a marriage of knowledge in these two disciplines that directed his professional life. He joined and became a board member of the International Academy of Gnathology, became a member of the Northwest Gnathological Restorative Seminar and served as a clinical associate professor at the University of Washington for 10 years. As he grew in knowledge and experience, Ralph realized that he had far more to offer his profession, so he



*Ralph Stenberg*

unselfishly assumed the mentor's responsibility for several study clubs in the Northwest.

Presently, he is a mentor of four study clubs that teach both restoration excellence and the need for greater knowledge of occlusion. His passion for dentistry has been documented in his numerous lectures to this Academy as well as many others. The common theme of excellence in these presentations define this individual.

There has been one common thread in the professional excellence displayed by clinicians such as Dr Ralph Stenberg. This is early involvement with the American Academy of Gold Foil Operators. Ralph has been a member of this Academy for the past 42 years. On behalf of the Academy, it gives me great pleasure to present the 1998 Clinician of the Year Award to Dr Ralph Stenberg.

RICHARD J HOARD

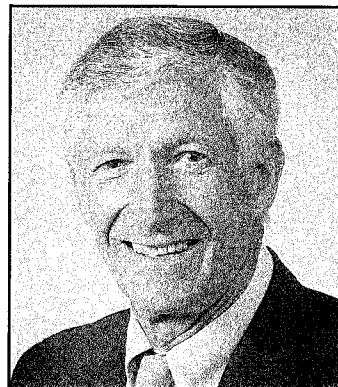
## Distinguished Member Award

Our recipient of the Distinguished Member Award this year is Dr Glenn Birkitt of Leesburg, Virginia. Glenn was born in Berkley Springs, West Virginia, and received his secondary education in Charles Town, West Virginia. Like many high school graduates, he didn't have a great sense of direction in his life, so he easily succumbed to the siren song of the US Marine Corps recruiter. Isn't it amazing how our government always seems to provide simple answers to life's most complex questions? And so, predictably, while dodging bullets on the hills of Korea, Glenn rapidly developed a sense of direction.

He returned to the States intact, and enrolled in pharmacy school at West Virginia University. After graduation, Glenn worked as a pharmacist in Leesburg, Virginia. Now, Glenn has always been one who takes advice seriously, so after hearing his minister admonish the congregation to spend their lives doing what they didn't like to do, he promptly applied to, and was accepted into the fourth class at the new School of Dentistry at West Virginia University. His pharmacy degree afforded him the luxury of financing his dental education by selling drugs after clinic hours. In 1964 he graduated, and he and his wife JoAnn returned to Leesburg to start a practice and a family. From that point until now, his life has been remarkably unremarkable.

Perhaps the greatest albatross in Glenn's life has been the fact that he was Bill Harris's classmate in dental school. The two became inseparable friends. After graduation, Bill was practicing in Beckley, West Virginia, and eventually found his way into the George M Hollenback Operative Dentistry Seminar. Bill and Glenn always attended the football games at West Virginia University together, which ultimately resulted in one of the great turning points in Glenn's life. In perhaps one of the greatest diplomatic coups of all time, Glenn agreed to join the study club, in exchange for Bill agreeing to stop lecturing about gold foil after the football games into the wee hours of the morning. The study club experience ultimately became one of the great loves of Glenn's life. He was able to find an avocation in the midst of his vocation.

When I later came along into the study club, and met Glenn, I was instantly impressed with his



*Glenn Birkitt*

operating prowess. If you have watched Glenn operate at our Academy meetings, you know what I mean when I say that he has perhaps the strongest, yet gentlest, hands that you could imagine. I've spent enough time observing in his office, and nosing around in his laboratory, to know that his study club and Academy operations are not just for show—it's the same dentistry that he practices in his office every day.

Glenn's professional accomplishments are numerous. In addition to having served as president of our Academy, he has also served on the Executive Council of the Academy of Operative Dentistry. He is a past president of the George M Hollenback Operative Dentistry Seminar, and is currently vice president of that fun-loving group, the American Association of Dental Insultants. He is also active in his state and local dental societies, as well as past president of the Leesburg Lions Club, and an Elder in the Presbyterian Church.

Members of the Academy of Gold Foil Operators, it gives me great pleasure to bestow this year's Distinguished Member Award on a consummate person, an excellent dentist, a dear friend, and a pretty good gentleman farmer, Dr Glenn Birkitt.

CRAIG BRIDGEMAN

# DEPARTMENTS

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Dr Peter Triolo, Chair  
Department of Restorative Dentistry and  
Biomaterials, Suite 452  
6516 John Freeman Avenue  
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A full-time position, Division Director of Operative Dentistry, is available within the Department of Restorative Sciences starting 1 July 1999. Responsibilities include administration of the division, directing all clinical and preclinical educational activities relating to operative dentistry, and assisting faculty in developing a strong divisional research program. The successful candidate must have completed a post-graduate training program in operative dentistry with diplomate status being preferred. The candidate will be expected to have outstanding contemporary clinical skills, evidence of significant previous research and teaching experience, and be eligible for tenure at this institution. There is an opportunity for private practice or consulting one day per week. Salary will be commensurate with experience and credentials. Applications will be accepted until the position is filled. The University of Minnesota is an Equal Opportunity educator and employer. Curriculum vitae and letter of intent should be sent to:

Dr James R Holtan, Chair  
Search Committee and  
Department of Restorative Sciences  
9-176 Moos Health Science Tower  
515 Delaware St SE  
Minneapolis, MN 55455

## ANNOUNCEMENTS

### TUCKER INSTITUTE CLINICAL COURSE

A clinical course on conservative cast gold restorations mentored by Richard V Tucker will be given at the University of Washington 14-18 June 1999. Participants will prepare and seat at least four castings. Patients can be provided upon request. The course fee of \$2000 covers all lab fees including gold. For course information and/or registration contact Dr Dennis Miya at 206-244-1618; FAX 206-431-9800.



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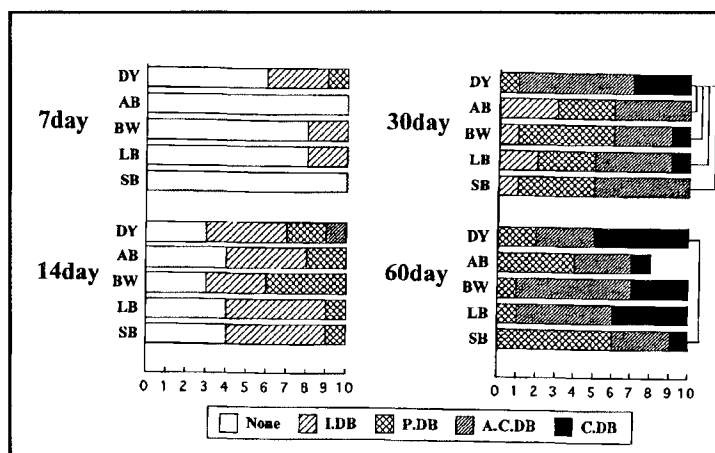
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## CORRECTION

Part of the following figure was inadvertently omitted from the article "Short-Term Reaction of Exposed Monkey Pulp beneath Adhesive Resins" by Kitasako and others, published in Volume 23(6):308-317. The graph was found on Page 311 (Figure 2). This is the figure as it should have been published. We regret the error.



## INSTRUCTIONS TO CONTRIBUTORS

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