OPERATIVE DENTISTRY

may-june 1999 • volume 24 • number 3 • 129-192

(ISSN 0361-7734)

OPERATIVE DENTISTRY

MAY-JUNE 1999 • VOLUME 24 • NUMBER 3 • 129-192

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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.

OPERATIVE DENTISTRY (ISSN 0361-7734) is published bimonthly for $60.00 per year in the USA (all other countries $70.00 per year) by University of Washington, Operative Dentistry, Health Sciences Bldg, Rm D-775, Seattle, WA 98195-7457. Periodicals postage paid at Seattle, WA, and additional mailing offices. **POSTMASTER:** Send address changes to: University of Washington, OPERATIVE DENTISTRY, Box 357457, Seattle, WA 98195-7457.

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EDITORIAL

A Dream of Preservative Health

At the stimulating American Association of Dental Schools meeting in Vancouver, BC, in March of this year, the need to follow the medical model seemed to be an axiom of the highest order. However, this has been our problem all along: We have been following medical's training methodology for far too long. It is time we moved away from treatment of disease and toward the philosophy of preservative health so aptly presented and described by Dr Ken Anusavice, Associate Dean for Research, University of Florida College of Dentistry. We here in the Pacific Northwest are blessed with a strong prevalence of preservative medicine being practiced by naturopathic doctors (NDs). Seattle even has within its city limits one of only two ND universities in the United States. My wife has been diligently trying to find a participating MD acceptable to our insurance program who believes and practices preservative medicine. When asked for a referral, one medical specialist told her, "There aren't any. We are all trained only to treat disease." This rather sad commentary on our medical profession is not uncommon. We should not be surprised at this, since the training received in medical and dental schools is, by far, geared only towards treatment of disease or problems. As was stated in an earlier editorial, this philosophy won't change for dentistry unless dentists are compensated for preserving the dental health of their patients.

There remains a glimmer of hope, however. Dedicated preservative health professionals like Dr Andrew Weil (MD) and Dr Ken Anusavice (DMD) are attempting to change the public's view of medicine and dentistry towards preservation of our health rather than treatment of disease. Dr Weil stated during one of his many television programs that he estimated only about 18% of medical patient visits were actually necessary. Preservative medicine would take care of the rest. Every day, young children are rushed to their pediatricians for antibiotics at the first sign of a runny nose. Tubes are placed in their ears as treatment for recurring ear infections. I shudder to think how abusive we have become towards the immune systems and bodies of our little people. Is it for convenience or simply a lack of knowledge that facilitates this behavior? A solid case could be made for either or both of these reasons depending upon the circumstances. As for myself, I am totally dedicated to maintaining the level of health that prevents a disease process from starting rather than treating it, once it is present. It is exciting to dream of a dental practice where only those who were committed to preserving their dental health could qualify as patients. What a feeling of satisfaction it would be to see children grow into adulthood without dental disease! While mentally exhilarating, it could be financially disastrous. It would seem logical for insurance to welcome the opportunity to pay for preservative treatment, for almost everyone would benefit—the patient because of continued good health, the employer because of fewer lost work hours, and the insurance company because of fewer claims for expensive treatment. Using this philosophy, the most threatened segment of our society would be the powerful drug companies whose main mission is to sell drugs for combating disease after it occurs.

Will conventional medicine and dentistry make dramatic advances toward preservative health in the near future? Probably not. As long as treatment of disease is the major focus of medical and dental schools, that is what will be practiced. However, we in the dental profession need to avoid looking upon the medical model as the epitome for teaching our students. We can certainly do better than that!

RICHARD B McCOY
Editor

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ORIGINAL ARTICLES

Effect of Cutting Instruments on Permeability and Morphology of the Dentin Surface

T SEKIMOTO • G D DERKSON • A S RICHARDSON

Clinical Relevance
Dentin bonding agents may have their effectiveness reduced when the dentin has been cut with diamond burs.

SUMMARY

There are major differences in morphological detail after cutting the dentin surface among the methods commonly used to prepare dental cavities. The purpose of this study was to compare dentin permeability and the morphology of the dentin surfaces prepared with diamond and carbide steel burs after etching with 6% citric acid. Twenty-four freshly extracted human third molars were sectioned, mounted on plexiglass, and connected to the dentin-permeability measuring apparatus. The permeability of dentin was measured by fluid filtration and expressed as hydraulic conductance. There were two study groups of 12 teeth. Each tooth had one occlusal cavity preparation prepared but utilized three depths: the original was prepared just into the dentin, the second 0.5 mm deeper than the first, and the third 0.5 mm deeper than the second. One group had the first cavity prepared with a diamond, the second deepened using a steel bur, then the third depth was made by use of the diamond. The other group had the first cavity preparation prepared with a steel bur, deepened 0.5 mm again using a diamond, then deepened again using a steel bur. Dentin permeability was measured after cavity preparation, then after 2 minutes of acid etching. Analysis of variance and Duncan's multiple range test were used to establish whether differences were significant at the 0.05 confidence level. Prepared and acid-etched surfaces were characterized using a scanning electron microscope to identify any differences between the two groups. After acid etching with 6% citric acid, the permeability of dentin cavities prepared with diamond burs was significantly less than the permeability of cavities prepared with carbide steel burs. After etching, there were differences in the appearance of the dentin surfaces prepared with diamonds

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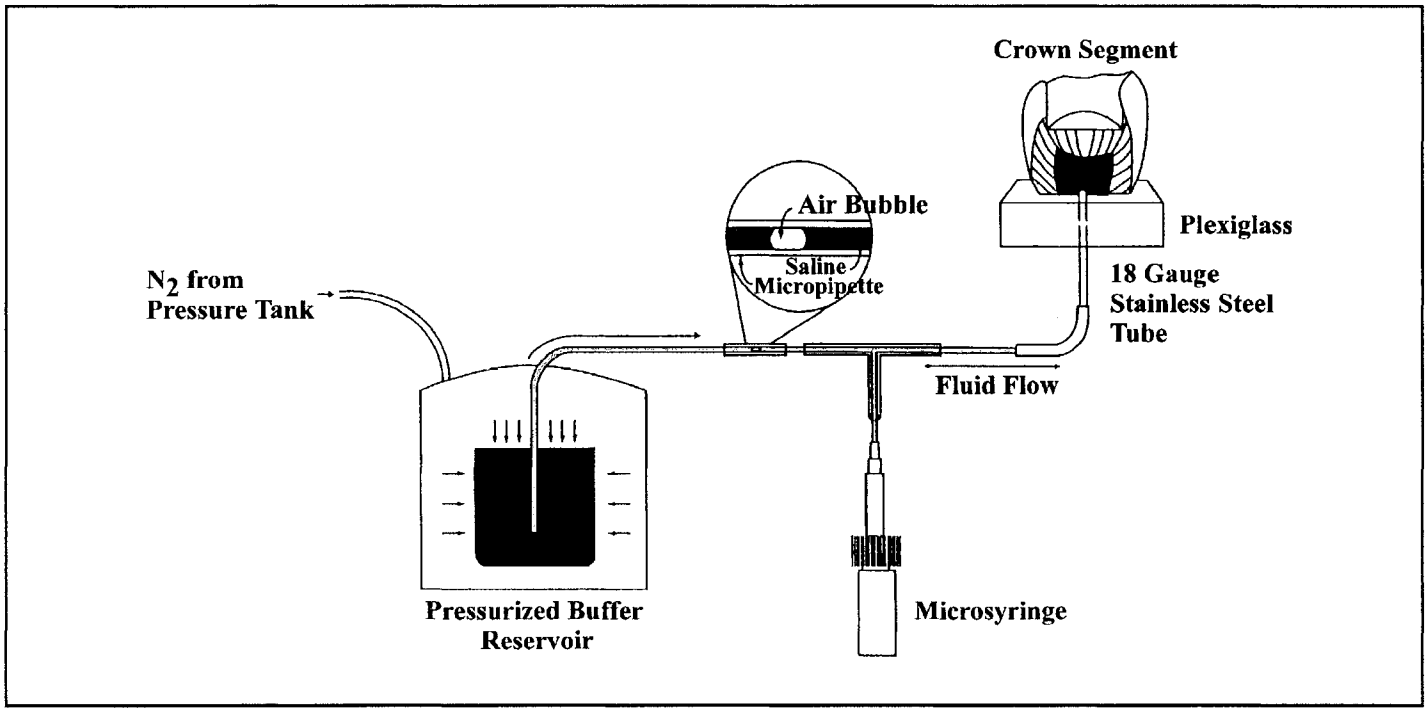

Figure 1. *Method used to quantitate dentin permeability. Nitrogen gas at a pressure of 15 psi was applied to a pressure reservoir containing a plastic beaker of phosphate buffered saline containing 0.2% fluorescein dye. The measurement of the movement of the air bubble in the micropipette per unit time quantitated permeability for each crown segment in µl/min. Tooth crown is viewed in cross section to show relation of cavity preparation to pulp.*

and steel burs. Dentin bonding agents may have their effectiveness reduced when placed following cavity preparation by use of a diamond.

INTRODUCTION

Following cavity preparation the enamel and dentin surfaces are generally covered with a thin layer of debris, commonly referred to as the smear layer. The production of a smear layer on dentin during restorative procedures establishes a barrier that can protect the dentin and the pulp from a potential irritant. In contrast, many investigators have reported that this debris can prevent bonding of a dental adhesive agent. Several dentin bonding systems have advocated smear layer removal, since it does not provide good mechanical support for adhesion of restorative materials. Hydrophilic primers penetrate moist dentinal tubules to enhance bonding to dentin. Since moist dentin is preferred in most dentin bonding systems, creating a smear layer that is readily removed would be desirable. Removal of the smear layer by acid conditioning increases the permeability of dentin which, under some conditions, must be regarded as a liability (Pashley, Michelich & Kehl, 1981). Measuring dentin permeability following acid etching to remove the smear layer could indicate whether it was readily removed.

On the other hand, rotary cutting instruments could create smear layers of varying resistance to acid removal. There are major differences in morphological detail after cutting the dentin surface among the methods commonly used to prepare dental cavities (Brännström, Glantz & Nordenvall, 1979). Significant differences were noted between burs and stones, though different speeds, with and without coolant, produced no notable differences (Scott & Wyckoff, 1946). The effect of rotary instrumentation on dentin permeability, after cutting with a diamond disk, carbide fissure bur, or diamond bur, was similar (Boyer & Svare, 1981). However, it has not been clearly shown if there are differences in dentin permeability following attempts to remove the smear layer by acid etching. The purpose of this study was to compare dentin permeability and the morphology of the dentin surface after a 2-minute etch with 6% citric acid on the dentin surfaces prepared with diamond burs and carbide steel burs.

METHODS AND MATERIALS

Measurement of Dentin Permeability

The work described in this paper was based on an in vitro method (Derkson, Pashley & Derkson, 1986). Twenty-four freshly extracted human unerupted third

molars that had been stored in normal saline containing 0.2% sodium azide were sectioned at the cementoenamel junction with a low-speed diamond saw (Isomet 11-1180, Buehler Ltd, Lake Bluff, IL 60044) to provide crown segments. The coronal pulp tissue was removed with a cotton forceps. Pieces of plexiglass had center holes drilled just large enough to accept 18-gauge stainless steel tubes, which were inserted so that the end was flush with the surface of the plexiglass. The metal tubes were sealed in place with cyanoacrylate. The crown segments were mounted on the plexiglass with cyanoacrylate and connected to the dentin-permeability measuring apparatus (Figure 1).

Phosphate-buffered saline containing 0.2% fluorescein dye was introduced into the pulp chambers of the crown segments at a constant pressure of 15 psi, a pressure similar to that used with the air-pressure technique. The dye assisted the visualization. The measurement of the movement of the air bubble in the micropipette per unit time quantitated permeability for each crown segment in µl/min.

There were two study groups of 12 teeth. After bonding to the Plexiglas, each tooth had an occlusal cavity preparation prepared to a depth of 0.5 mm into dentin. The second cavity preparation was prepared 0.5 mm deeper than the first, and the third was prepared 0.5 mm deeper than the second. Group 1 had the first preparation prepared with a high-speed handpiece using a #310 diamond stone (Shofu Dental Products, Kyoto, Japan); the preparation was then deepened 0.5 mm using a #36014 steel bur (Busch & Co, Engelskirchen, Germany) in a low-speed handpiece. The pulpal wall was then deepened another 0.5 mm using the same diamond used initially. Group 2 had the first cavity preparation prepared with a steel bur, deepened 0.5 mm using the #310 diamond, and then deepened 0.5 mm again using the original steel bur.

Dentin permeability was measured after mounting (error measurement), after each cavity preparation depth change, and after acid etching with 6% citric acid for 2 minutes. As the maximum rate of fluid filtration was assigned a value of 100%, the percent of change in fluid flow rate over the course of the experiments was reported. Each tooth was its own control, and actual measurements were converted to a percentage value with the highest value for a series of measurements of a crown segment designated 100%. Analysis of variance and Duncan's multiple range test were used to establish whether differences were significant at the 0.05 confidence level.

Scanning Electron Microscopy (SEM)

Dentin disks were cut from extracted human unerupted third molars perpendicular to the long axis of the teeth with a diamond disk (Komet Diamond disk 918B, Komet Inc, Lemgo, Germany) for SEM examination.

The cavity side of the dentin was divided into four areas, and each area was treated as follows: (1) prepared with a low-speed handpiece using a #36014 steel bur at 40,000 rpm; (2) prepared with a high-speed handpiece using a #310 diamond at 380,000 rpm; (3) prepared as (1) and etched with 6% citric acid for 2 minutes; (4) prepared as (2) and etched with 6% citric acid for 2 minutes.

Each area was characterized using SEM, and representative photomicrographs were obtained to examine differences in the appearance of each area.

RESULTS

Apparent dentin permeability of cavities prepared with diamond and steel burs as the percent of fluid

Percent of Fluid Flow Rate of Cavities Prepared with Diamond Burs and Steel Burs

	Diamond	Etch 1	Steel	Etch 2	Diamond	Etch 3
Group 1	1.32 ± 0.14	10.08 ± 1.06	4.89 ± 0.50	100.00	7.52 ± 1.18	38.13 ± 2.21

	Steel	Etch 1	Diamond	Etch 2	Steel	Etch 3
Group 2	1.38 ± 0.14	40.20 ± 1.64	5.72 ± 0.75	19.92 ± 1.31	4.06 ± 0.27	100.00

Values connected by lines are not statistically different at $P < 0.05$.

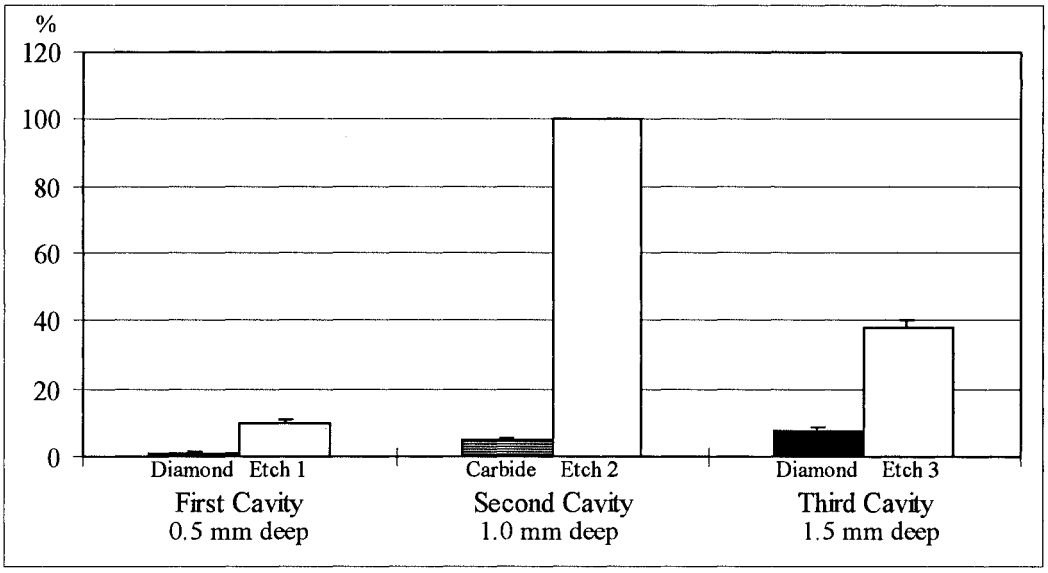

Figure 2. *Effect of acid etching on dentin permeability of the three cavities (Group 1)*

flow rate are shown in the table. Dentin permeability was measured after cavity preparation and after acid etching (Etch 1, Etch 2, Etch 3). The maximum rate of fluid filtration, which was assigned a value of 100%, was Etch 2 in Group 1, and Etch 3 in Group 2. The horizontal lines connecting groups indicate that these groups are not statistically different from each other. Before acid etching, the dentin permeability after the first cavity preparation was very low (1.32 ± 0.14%, 1.38 ± 0.14%), and there were no statistical differences among the first cavity preparation, the second, and third, both in Group 1 and in Group 2. After acid etching with 6% citric acid, the permeability of cavities prepared with the diamond bur was statistically significantly less ($P < 0.05$) than the permeability of cavities prepared with the steel bur both in Group 1 and in Group 2. The expectation is that, as the cavities get closer to the pulp, the permeability after etching with 6% citric acid would become greater. When a carbide bur was used for the last and deepest cut, the permeability was 100% (Figure 3). However, when a diamond bur was used for the last and deepest cut, permeability only reached 38% of the maximum that occurred after cutting with the carbide bur for the second cavity (Figure 2).

Representative SEM photomicrographs are shown in Figures 4 to 7. Figure 4 demonstrates a surface of the dentin after preparation with a carbide steel bur. Figure 5 demonstrates a surface of the dentin after preparation with the diamond bur. Before acid etching, little difference was observed between the surfaces prepared with the steel bur and the diamond bur except the dentinal grooves left by the diamond bur. Both of the surfaces were completely covered with a fairly thick smear layer, and the tubule apertures were not discernible because they were occluded. Figure 6 demonstrates a surface of the dentin after preparation with a steel bur and acid etching. Figure 7 demonstrates a surface of the dentin after preparation with a diamond bur and acid etching. After acid etching, a significant difference was observed between the surfaces prepared with a steel bur and a diamond bur. Most of the smear layer of debris has been removed and the tubule apertures are clearly visible on the dentin prepared with a steel bur. In contrast, the surface is still covered with a thin smear layer on the dentin prepared with a diamond bur, and the positions of the tubules are barely discernible.

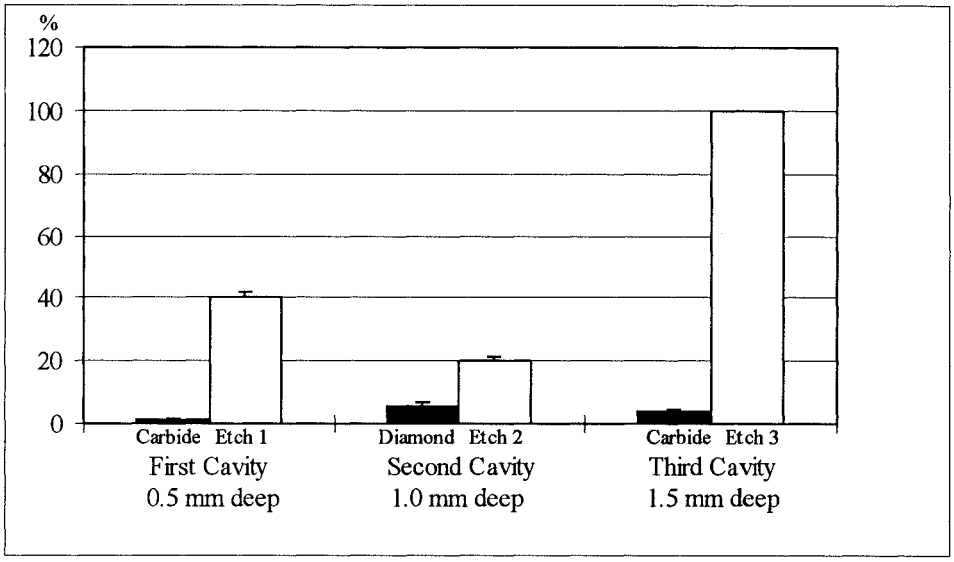

Figure 3. *Effect of acid etching on dentin permeability of the three cavities (Group 2)*

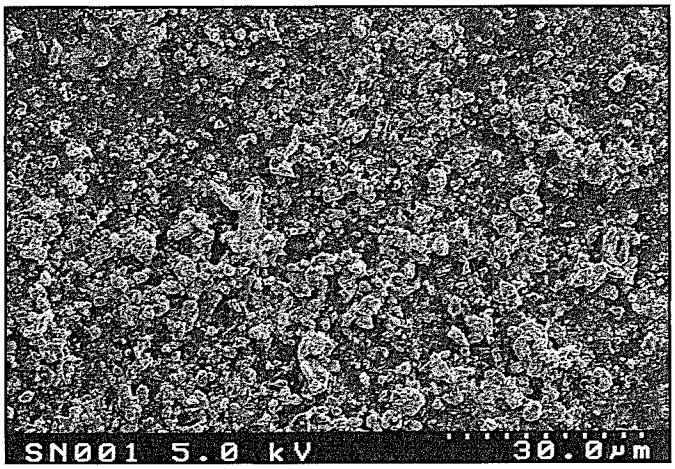

Figure 4. *SEM of dentin surface prepared with steel bur. The surface is completely covered with a fairly thick smear layer, and the tubule apertures are not discernible.*

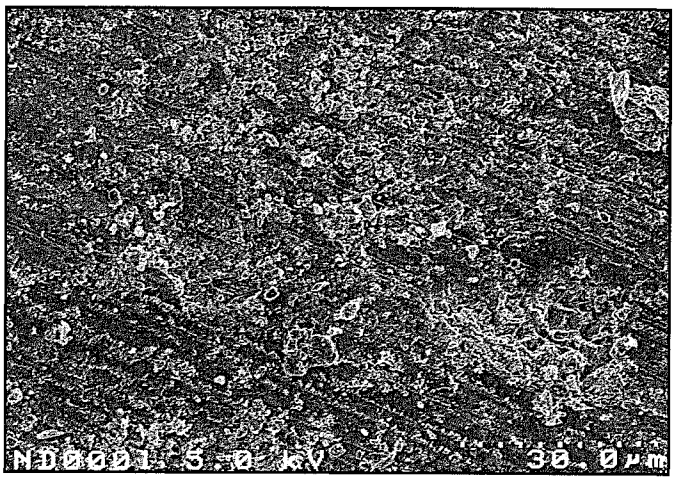

Figure 5. *SEM of dentin surface prepared with a diamond bur. Before acid etching, little difference was observed between the surfaces prepared with steel bur and diamond bur except the dentinal grooves left by the diamond bur.*

DISCUSSION

Dentin Permeability

The production of a smear layer on dentin during restorative procedures establishes a protective diffusion barrier. It has been reported that 86% of the total resistance to fluid movement across dentin in vitro is due to the presence of the smear layer (Pashley, Livingston & Greenhill, 1978). According to the study in which the dentin permeability was measured with the same apparatus and method, when the smear layer was intact, dentin permeability was 6.32 ± 2.5% in relation to the acid-etched values of the cavity preparations (Pashley & Depew, 1986). In accordance with the reports, the results of this study demonstrated that the dentin permeability after cavity preparation was very low, independent of the cutting instruments and the cavity depth (Min 1.32 ± 0.14%, Max 7.52 ± 0.14%). Removal of the smear layer by acid-etching increased the permeability of dentin, which, under some conditions, must be regarded as a liability (Pashley & others, 1981). Acid etching of dentin is recommended with many dentin bonding systems to remove the smear layer and to permit bonding directly to the dentin matrix. Many

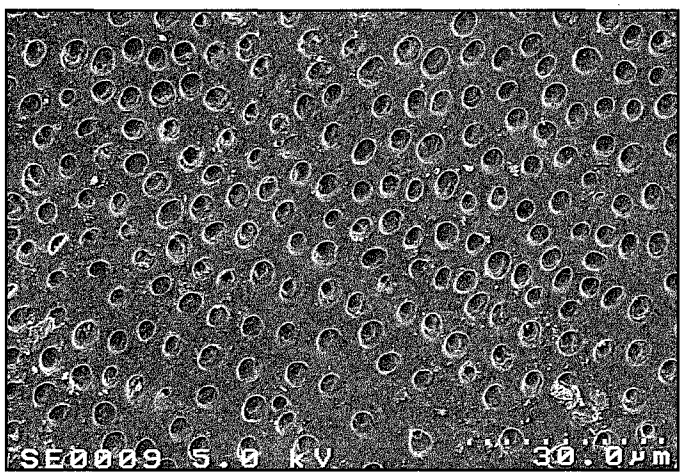

Figure 6. *SEM of dentin surface prepared with steel bur and etched with 6% citric acid for 2 minutes. Most of the smear layer of debris has been removed and the tubule apertures are clearly visible.*

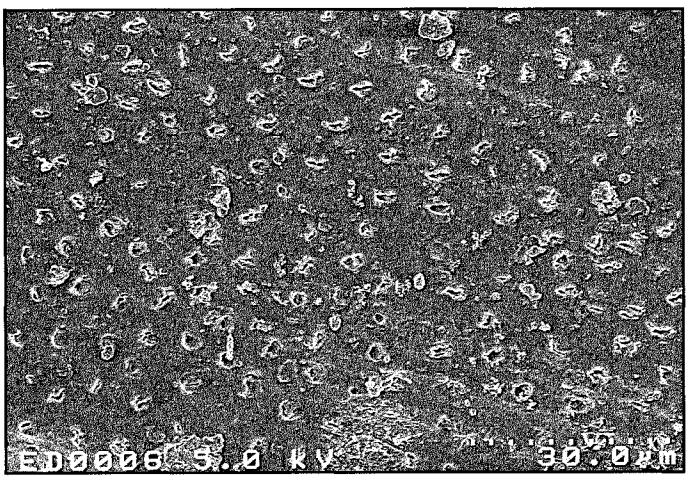

Figure 7. *SEM of dentin surface prepared with a diamond bur and etched with 6% citric acid for 2 minutes. The surface is still covered with a thin smear layer, and the positions of the tubules are barely discernible.*

acid-etching protocols have been suggested as suitable for dentin conditioning (Gwinnett, 1984), including 37% phosphoric acid for 15 seconds (Brännström, 1984), 10% citric acid for 60 seconds, 25% tannnic acid for 15 to 60 seconds (Bitter, 1989; Takahashi & others, 1993), 10% polyacrylic acid for 20 seconds (García, 1992), and 10% maleic acid for 10 seconds (Swift & Triolo, 1992). In this study, all cavities were etched with 6% citric acid to remove the smear layer as opposed to using a commercial acid as provided by the manufacturer. We wished to use methods and materials that had been standardized by Pashley and others (1981). This allows the readers the ability to make comparisons. All of Pashley's work has shown that using 6% citric acid is very good for removing the smear layer. Certainly, using phosphoric acid may make a difference, but that is the subject of our next study. Acid etching with 6% citric for 2 minutes led to a very large, statistically significant increase in dentin permeability (Pashley & Depew, 1986). The results of this study demonstrated that the permeability of the dentin cut with steel burs clearly increased by acid etching, but the permeability of the dentin cut with diamonds was not significantly different after acid etching except the third cavity in Group 1. In coronal dentin the number of tubules per unit area reaches its maximum close to the pulp. Also the diameter of tubules increases as they approximate the pulp. The total tubular surface near the DEJ is approximately 1% of the total surface area of dentin (Pashley, 1983), whereas close to the pulp chamber the total tubular surface area may be nearly 45%. Thus from a clinical standpoint it should be recognized that dentin beneath a deep cavity preparation is much more permeable than dentin underlying a shallow cavity (Cohen & Burns, 1991).

Our results demonstrated that the increments of dentin permeability associated with depth were not found in the cavities that were etched after preparation with diamond burs. In contrast, the dentin permeability of the cavities that were etched after preparation with the steel bur increased clearly in proportion to the depth of the cavity. These results suggested that the type of cutting instrument has an influence on any increase of dentin permeability by acid etching. The permeability of dentin after cutting with a diamond disk, carbide fissure bur, or diamond bur has been shown to be similar (Boyer & Svare, 1981). The influence of the smear layer on the permeability was about the same for all rotary instruments and did not depend on type or speed (Dippel, Borggreven & Hoppenbrouwers, 1984). However, differences in dentin permeability following attempted smear layer removal by acid etching are not clear. A comparison between a diamond bur and a steel bur was made in this study. Following acid etching with 6% citric acid, the permeability of cavities prepared with the diamond bur was statistically significantly less ($P < 0.05$) than the permeability of cavities prepared with the steel bur. This indicated that the smear layer was not readily removed when the cavity was prepared with a Shofu #310 diamond bur.

Scanning Electron Microscopy (SEM)

Dentin disks were cut from extracted human teeth perpendicular to the long axis of the teeth with a diamond disk for SEM examination. The cavity side of the dentin surface was divided into four areas to examine the difference in appearance between the cutting instruments. The center of the disk has fewer tubules per unit surface area than over the pulp horns (Pashley & others, 1987). Since the central dentin was not the subject for the SEM examination, the differences in the number and diameter of the tubules of each area were disregarded in this study. Before acid etching, little difference was observed between the surfaces prepared with a steel bur and a diamond bur except the dentinal grooves left by the diamond bur. Both of the surfaces were completely covered with a fairly thick smear layer, and the tubule apertures were not discernible because of this occlusion. According to the study on the smearing effect of different types of rotary instruments on dentin using Ash SC fissure 1, SSW plain fissure FG 57, and diamond stone Horico K 3/3, it was concluded that the degree of coverage of the smear layer depended on the type of rotary instrument (Dippel & others, 1984). Their findings showed a dependence on the bur speeds; there were no differences between the instruments over 25,000 rpm. The dentin was prepared with a diamond bur at about 380,000 rpm and a carbide steel bur at 40,000 rpm in this study; therefore, little difference was observed on the dentin surfaces between the instruments.

After acid etching, a significant difference was observed in the appearances of the surfaces between surfaces prepared with the carbide steel bur and diamond. Surfaces were still covered with a thin smear layer on the dentin prepared with the diamond bur, and the positions of the tubules were barely discernible, caused by plugging of the tubules. In contrast, most of the smear layer was removed and the tubule apertures were clearly visible on the dentin prepared with the carbide steel bur.

The smear layer produced by the steel bur was readily removed by acid etching, compared to the smear layer produced by the preparation with the diamond bur. The quality and quantity of smear layer appeared to be influenced by the type of instrumentation. Diamond instruments produce

extensive, well-retained, amorphous surface layers (Brännström, Glantz & Nordenvall, 1979). Diamond burs are usually used for cavity preparations in Japan, which is not the usual way North American dentists work. It appears from this study that when #310 diamond burs are used to cut teeth, the resultant smear layer is resistant to acid removal. This has been shown by permeability measurement and electron microscopic examination. Whether having a smear layer that is resistant to acid dissolution has an effect on bond strength is the question. This study does not answer the question; however, it suggests that it needs to be studied. There are many processes in dentistry where diamond burs are used to cut dentin. In these same situations dentists use dentin bonding. It is well accepted that smear layer removal is required to successfully bond to dentin. This study suggests that the smear layer created by cutting with a #310 diamond bur is not completely removed. This could affect bond strength of the restorations. It appears that the type of cutting instrument has an influence on dentinal bond strength.

CONCLUSIONS

The results of this study permit the following conclusions:

1. After acid etching with 6% citric acid, the permeability of cavities prepared with Shofu #310 diamond burs was significantly less than the permeability of cavities prepared with #36014 Busch carbide steel burs.
2. The surface was still covered with a thin smear layer on the dentin prepared with a diamond bur regardless of etching.
3. Etching dentin cut with #310 diamond burs may result in the incomplete removal of the smear layer, particularly from the dentinal tubule orifice. This could affect bond strength of the restoration.

(Received 12 November 1997)

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Marginal Adaptation of Heat-pressed Glass-Ceramic Veneers to Dentin in Vitro

M CHRISTGAU • K-H FRIEDL
G SCHMALZ • U RESCH

Clinical Relevance
High-viscosity composite resin cements used with their matching bond system provide good marginal adaptation of ceramic veneers both to enamel and to dentin.

SUMMARY

The purpose of the present study was to examine the marginal adaptation of ceramic veneers to dentin at the cervical margins and to enamel at the palatoincisal margins using four dual-curing composite resin cements of different viscosity with their corresponding dentin bonding systems. Thirty-six caries-free human maxillary incisors were prepared for facial ceramic veneers with cervical cavity margins located in dentin. Heat-pressed glass-ceramic veneers (IPS Empress) were inserted adhesively using one of the following luting systems: Sono-Cem (SC) with EBS; Variolink Ultra (VU), Variolink High Viscosity (VHV), and Variolink Low Viscosity (VLV) with Syntac. Both the cervical and the palatoincisal margins of the veneers (tooth/composite resin cement interface and ceramic/composite resin cement interface) were evaluated before and after thermocycling and mechanical loading (TCML) by quantitative margin analysis under a scanning electron microscope (SEM) using an image analysis system. Microleakage was assessed by dye penetration after TCML. Before TCML, SC and VU showed statistically significantly fewer marginal gaps than VHV and VLV. After TCML, SC, VU, and VHV revealed significantly fewer marginal gaps than VLV. TCML had a statistically significant influence on marginal gap formation at both the dentin and enamel margins. After TCML, the percentage of marginal gaps was not significantly different at the cervical dentin than at the palatoincisal enamel margins. Cervical dye penetration after TCML showed no statistically significant differences

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in microleakage among the four luting systems. In conclusion, this in vitro study showed that similarly favorable marginal adaptations of ceramic veneers to dentin and enamel can be achieved using Sono-Cem, Variolink Ultra, or Variolink High Viscosity with their corresponding dentin bonding systems.

INTRODUCTION

Ceramic veneers are a well-established treatment method for conservative esthetic restoration of malformed, discolored, or malaligned anterior teeth (Calamia, 1985,1989; Chalifoux, 1994; Dunne & Millar, 1993; Jordan, Suzuki & Senda, 1989; Sheets & Taniguchi, 1990). The recommended superficial preparation within the enamel and adhesive luting (Roulet & others, 1989) facilitates a restoration with minimal loss of healthy tooth substance. The potential for pulpal involvement is significantly reduced compared to crown preparation techniques (Dunne & Millar, 1993). Supragingival veneer margins avoid the risk of irritation to periodontal tissues (Coyne & Wilson, 1994). Short- and long-term clinical studies in highly selected patient groups (Calamia, 1989; Coyne & Wilson, 1994; Dunne & Millar, 1993; Jordan & others, 1989) have shown that ceramic veneer restorations, which were bonded to enamel only, are a reliable and effective long-term procedure for the conservative treatment of anterior dentitions, although scanning electron microscope assessments indicated that ideal marginal adaptation is difficult to achieve (Coyne & Wilson, 1994). In a retrospective 5-year clinical study by Dunne and Millar (1993), 83% of 315 labial ceramic veneers were categorized as being clinically satisfactory at the final recall. Increased problem and failure rates were associated with veneers placed on existing restorations, where tooth surface loss had occurred prior to treatment, and where inappropriate luting agents were employed.

Currently, ceramic veneers are mainly recommended for cavity preparation margins located in enamel providing reliable marginal integrity (Chalifoux, 1994; Schmalz, Federlin & Geurtsen, 1994). However, even with veneer preparation margins located coronally to the cementoenamel junction, Nattress and others (1995) found a high risk for dentin exposure at the cervical margins. Furthermore, therapy of advanced periodontal disease often results in postoperative gingival recession with exposed root surfaces and wide interproximal spaces causing esthetic and phonetic impairments. When dealing with intact teeth, this poor esthetic situation might be overcome by ceramic veneers with margins extended onto dentin instead of using full crown restorations, which suffer from a higher risk of pulpal and gingival damage. Previous in vitro studies (Sim & others, 1994; Tjan, Dunn & Sanderson, 1989; Zaimoglu, Karaagaclioglu & Üctasli, 1992) have shown that the dentin-composite resin cement interface of veneers with cervical margins located in dentin was significantly more susceptible to microleakage than all-enamel preparations. So far, this has been true independent of the use of dentin bonding agents (Sim & others, 1994). Forces caused by thermal stress and polymerization shrinkage of the composite resin cements are regarded as the main causes for failure of the weak link between composite resin cement and dentin. The resulting microleakage is considered a risk factor for marginal staining, postoperative sensitivity, and recurrent caries (Castelnuovo, Tjan & Liu, 1996; Tjan & others, 1989; Zaimoglu & others, 1992). However, recently a considerable increase in bond strength and quality of marginal adaptation has been achieved with the latest generation of bonding systems, which approximate the bond strength of composite resin to acid-etched enamel (Eick & others, 1997; Perdigao & others, 1994).

Previous in vitro studies (Schmalz, Federlin & Reich, 1995; Thonemann & others, 1994) on ceramic inlays with margins located in dentin or enamel found less marginal gap formation and microleakage after using highly filled composite resin cements. So far, there is no report in the literature assessing the influence of the viscosity of composite resin cements on the marginal adaptation of ceramic veneers to dentin. For this reason, the purpose of the present in vitro study was to examine the influence of the viscosity of different dual-curing composite resin cements on the marginal adaptation and integrity of heat-pressed glass-ceramic veneers to dentin at the cervical margin and to enamel at the palatoincisal margin.

METHODS AND MATERIALS

Preparation and Veneer Fabrication

Thirty-six caries-free human maxillary incisors were stored in a 0.1% thymol solution at room temperature for less than 4 weeks after extraction. Then the teeth were stored in 0.9% saline for at least 1 week in a refrigerator (4 °C). The teeth were scaled, cleaned with pumice, and the apices were sealed with gutta percha, after which they were embedded in a polymethylmethacrylate resin (Pattern Resin, GC Dental, Tokyo, Japan) up to 5 mm below the cementoenamel junction (CEJ) in molds matching the clamps of the thermocycling machine. One clinician prepared all teeth for facial ceramic veneers using diamond burs (Brasseler, Lemgo, Germany). First, horizontal depth-orientation grooves were made using a specially designed 0.5 mm depth-gauge bur providing a well-defined reduction of the enamel

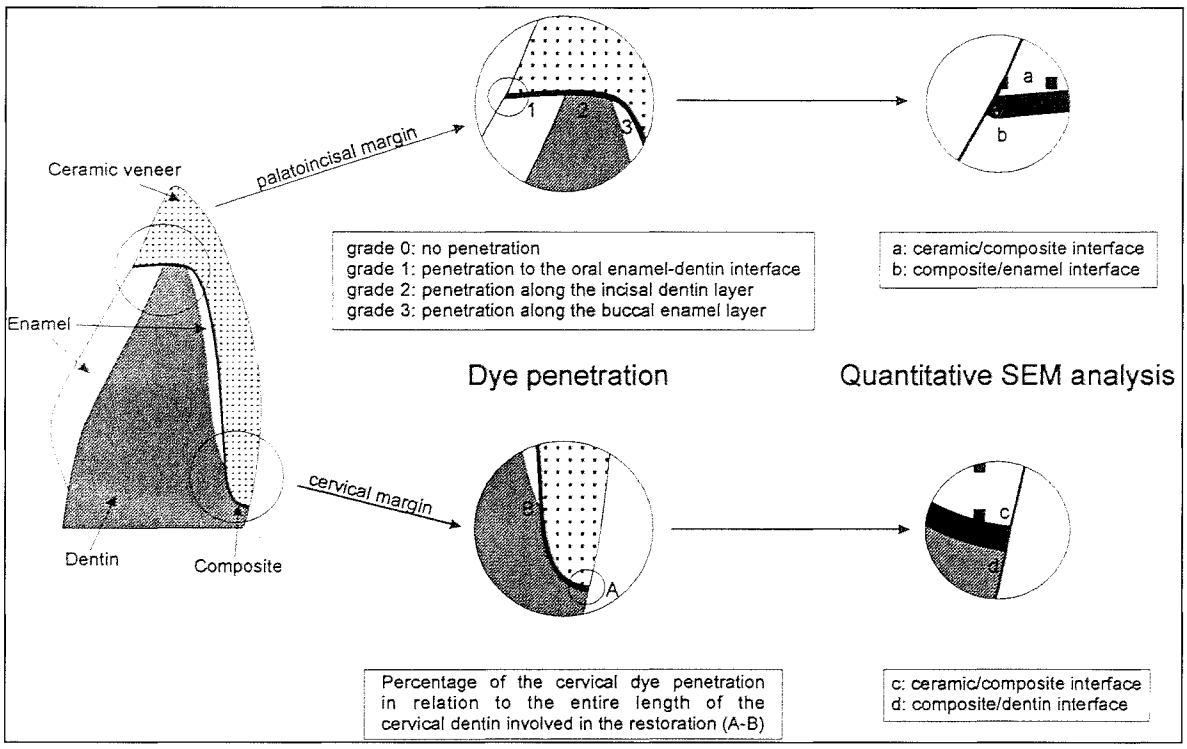

Figure 1. Extension of the veneer preparation, the evaluation method for dye penetration, and the interfaces evaluated by SEM

layer. Using a chamfered-end, parallel-sided diamond bur, an even reduction of the buccal surface with chamfered finishing lines at all margins was achieved. The incisal edge was reduced and the preparation was extended about 1 mm onto the palatal surface (Figure 1). Interproximally, the preparation was extended about halfway into the interproximal contact area. On the facial side, the cervical preparation was extended 2 mm beyond the CEJ, exposing the root dentin. All other margins were located within enamel. The preparations were finished using a matching diamond finishing bur (40 µm particle size, Brasseler). Impressions were taken using a polyether material (Impregum, ESPE, Seefeld/Oberbay, Germany) and customized plastic trays. Following impressions, the teeth were stored again in 0.9% saline at 37 °C until completion of the ceramic veneers. All veneers were fabricated by one dental technician using a high-strength, fine-grained, and leucite-reinforced heat-pressed glass-ceramic material (IPS Empress, Vivadent, Schaan, Liechtenstein) according to the manufacturer's instructions. Because of the in vitro situation, individual coloring was omitted. After drying the teeth, the veneers were tried in. The fit was controlled using a binocular light microscope at X10 magnification. The internal surfaces of the veneers were etched with 5% hydrofluoric acid (IPS Etching Gel, Vivadent) for 60 seconds, rinsed with tap water, and air dried. Then the veneers were silanated with a coat of Monobond S (Vivadent).

After thorough cleaning of the teeth, the cavity preparations were etched with 37% phosphoric acid (MiniTip, ESPE) for 30 seconds, rinsed with water spray for 30 seconds, and dried thoroughly. Then the 36 teeth were randomly assigned to four groups of nine teeth each. The ceramic veneers were inserted using the following four different dual-curing composite resin cements with their corresponding dentin bonding agents according to the manufacturers' instructions (Table 1): (1) Sono-Cem (SC) with the ESPE Bonding System; (2) Variolink Ultra (VU) with the Syntac bonding system (Vivadent); (3) Variolink High Viscosity (VHV) with the Syntac bonding system (Vivadent); (4) Variolink Low Viscosity (VLV) with the Syntac bonding system (Vivadent). The dentin bonding agents were applied to the exposed cervical dentin according to the manufacturers' instructions. Both the preparations and the inner surfaces of the veneers were then coated with the unfilled bonding resin of the dentin bonding system (Table 1), which was air thinned but not light cured. The dual-curing composite resin cements were

applied to the inner surface of the veneers, before the veneers were luted to the cavity preparations. Veneers, which were luted with VHV or VLV, were inserted by finger pressure, while veneers, luted with Sonocem or Variolink ultra, were inserted by the ultrasonic insertion technique (Noack & others, 1992; Peutzfeldt, 1994; Walmsley & Lumley, 1995) using the thixotropy of these highly filled composites. For this technique, an ultrasonic device (Cavitron, Dentsply Preventive Care Division, York, PA 17404) was used, consisting of a specially designed two-arm metal tip, which was covered with protective plastic. Excess resin was removed with a small metal spatula. The remaining luting material was covered with a glycerin gel and light cured from all directions for 2 minutes (Heliolux, Vivadent, Ellwangen, Germany). The curing light output was monitored using a light meter (Cure Rite, Caulk/Dentsply, Milford, DE 19963). The gross composite excess was removed immediately after insertion of the veneers using finishing diamonds (Composhape, Intensiv, Viganello-Lugano, Switzerland). Then the specimens were stored in 0.9% saline at 37 °C for 24 hours to improve the degree of polymerization of the chemically activated part of the dual-curing cements and to improve the visibility of subtle composite resin excess. The veneers were finished and polished 24 hours after cementation using finishing diamonds and flexible polishing disks (Sof-Lex, 3M Dental Products, St Paul, MN 55144). The teeth were stored again in 0.9% saline for 48 hours before further processing.

Thermocycling and Mechanical Loading (TCML)

In groups of six, the teeth were thermocycled, alternating between 5 °C and 55 °C for 5000 cycles. The dwell time at each temperature was 30 seconds. During the 5000 thermal cycles, 500,000 mechanical load cycles were performed on the incisal edge in the direction of the tooth axis with a frequency of 1.6 Hz and a load of 72.5 N.

Quantitative Margin Analysis

Before and after TCML, facial and palatal impressions were taken using customized trays and a vinyl polysiloxane impression material (Permagum, ESPE). Replications were made using a bisphenol A epoxy resin (Araldit, Ciba Geigy, Wehr, Germany). They were gold sputtered and the integrity of the cervical and the palatoincisal margins was examined using a scanning electron microscope (Stereoscan 240, Cambridge Instruments, Nußloch, Germany) at X200 magnification. The quantitative margin analysis was performed at the tooth/composite resin cement and the ceramic/composite resin cement interfaces at both the cervical and the palatoincisal margins (Figure 1) using an image analysis system (Videoplan, Kontron, Eching, Germany) according to the procedures described by Roulet and others (1989). Using one adjustment for each replica side in the SEM, the entire visible length of the cervical or the palatoincisal veneer margin was evaluated. The following criteria were used for the description of the marginal quality: (a) perfect margin: tooth/composite or composite/ceramic interfaces are completely smooth without any interruption of continuity; (b) marginal gap: tooth and composite or composite and ceramic are separated by a gap caused by adhesive or cohesive failure; (c) marginal imperfection: any kind of incontinuity of the tooth/composite interface or composite/ceramic interface but without a marginal gap, e g, marginal ceramic chipping, marginal enamel fracture, overhang or underfilled margin.

The absolute lengths of the different marginal qualities were transformed to percentages of the entire visible lengths of the cervical or palatoincisal margins.

Dye Penetration

After TCML, all tooth surfaces were covered with nail varnish up to 0.5 mm to the cervical and palatoincisal margins of the restorations. The interproximal veneer margins were also covered with nail varnish to prevent interference by dye penetration from these sides. The teeth were stored for 16 hours at 37 °C in a 0.5% basic fuchsin (Fluka, Buchs, Switzerland) solution. The teeth were sectioned (thickness: 300 µm) in a faciolingual direction using a water-cooled low-speed diamond saw (Microtome 1600, Leitz, Wetzlar, Germany). Both sides of each section were photographed at X8 magnification. Microleakages were evaluated in different ways at the cervical and palatoincisal margins (Figure 1) using an image analysis software (Videoplan, Kontron, Germany): (a) at the cervical margin, the length of the dye penetration was expressed as percentage of the entire length of the cervical dentin involved in the restoration; (b) at the palatoincisal margin, the extension of the dye penetration was graded according to the following criteria: Grade 0—no penetration; Grade 1—penetration along enamel up to the palatal enamel-dentin interface; Grade 2—penetration along the incisal dentin layer; Grade 3—penetration along the facial enamel layer.

Statistical Analysis

For both examination methods (SEM, dye penetration), the median values (with 25/75% percentiles) of nine replications were calculated for each product combination, margin, interface, and time. For each single tooth the maximum dye penetration of all

Table 1. Dentin Bonding Agents and Corresponding Composite Resin Cements

Group	Dentin Bonding Agent	Chemical Composition % by weight	Batch #	Composite Resin Cement (% by weight)	Insertion Technique	Batch #	Manufacturer
1 n=9	ESPE Bonding System (EBS)			Sono-Cem (SC) filler content: 77.3%	ultrasonic insertion technique	Base + Catalyst 111	ESPE, Seefeld/ Oberbay, Germany
	Primer	50% HEMA, 10%MMC, 40% water	001				
	Bond	73% Bis-Methacrylate, 7% HEMA, 17% MAM, initiators and stabilizers	001				
2 n=9	Syntac			Variolink Ultra (VU) filler content: 79%	ultrasonic insertion technique	Base 660017	Vivadent, Schaan, Liechtenstein
	Primer	25% TEGDMA, 4% maleic acid, 41% Di-methyl-ketone, 30% water	618657			Catalyst 602374	
	Adhesive	35% Poly-EGDMA, 10% glutaraldehyde, 55% water	701046				
	Heliobond	60% BIS-GMA, 40% TEGDMA	613764				
3 n=9	Syntac	same	same	Variolink High Viscosity filler content: 76%	finger pressure	Base 614052	Vivadent
						Catalyst 616853	
4 n=9	Syntac	same	same	Variolink Low Viscosity filler content: 72%	finger pressure	Base 614054	Vivadent
						Catalyst 614243	

n = number of evaluated teeth. Chemical composition according to manufacturer. TEGDMA = tri-ethylene-dimethacrylate; HEMA = 2-hydroxyethyl-methacrylate; Poly-EGDMA = poly-ethylene-glycol-dimethacrylate; MMC = methacryl-magnesium-chelate; BIS-GMA = bis-phenol-glycidylmethacrylate; MAM = malonic acid-alkyl-methacrylate.

sections per tooth was considered for further evaluation describing the worst site of each tooth. Because of the nonnormal distribution of data and varying standard deviations, the statistical analysis was performed using the Mann-Whitney-U test for pairwise comparisons at a significance level of $\alpha = 0.05$ (SPSS/PC+, Ver. 5.01, SPSS Inc, Chicago, IL 60611). For testing the overall influence of factors, the levels of significance were adjusted to $\alpha^* = 1-(1-\alpha)^{1/k}$ (k = number of performed pairwise tests) using the error rates method (Miller, 1981).

RESULTS

SEM Analysis

Figures 2-5 and Table 2 show the SEM results and statistical analysis at each interface before and after TCML.

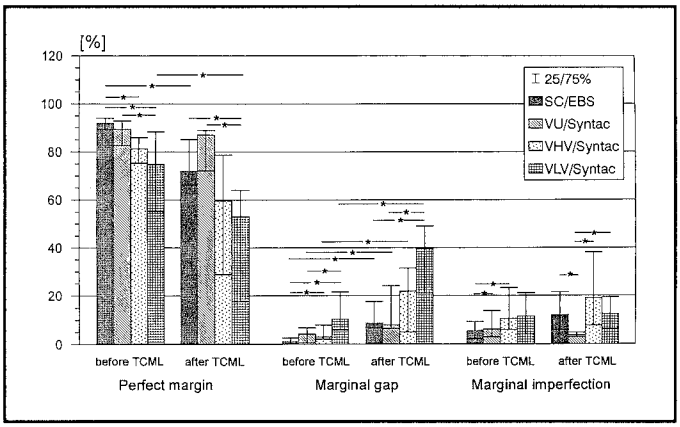

Figure 2. *Quantitative SEM margin analysis of the dentin/composite resin cement interface at the cervical margin: median (25/75%) percentage of perfect margin, marginal gap, and marginal imperfection before and after TCML. Significant differences (P ≤ 0.05) between medians are labelled with *. SC = Sono-Cem; EBS = ESPE Bonding System; VU = Variolink Ultra; VHV = Variolink High Viscosity; VLV = Variolink Low Viscosity.*

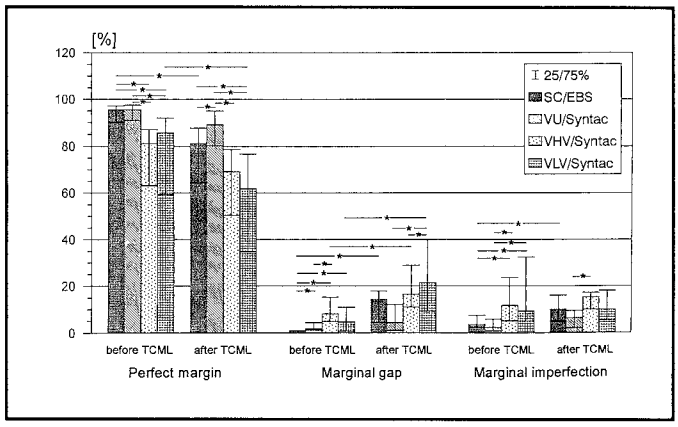

Figure 3. *Quantitative SEM margin analysis of the ceramic/composite resin cement interface at the cervical margin: median (25/75%) percentage of perfect margin, marginal gap, and marginal imperfection before and after TCML. Significant differences (P ≤ 0.05) between medians are labelled with *. SC = Sono-Cem; EBS = ESPE Bonding System; VU = Variolink Ultra; VHV = Variolink High Viscosity; VLV = Variolink Low Viscosity.*

Cervical Dentin/Composite Resin Cement Interface (Figure 2)

Before TCML, SC showed significantly fewer marginal gaps and significantly more perfect margins than VHV and VLV. VU revealed significantly fewer marginal gaps than VLV. After TCML, the percentage of marginal gaps increased significantly for all luting systems, while a significant decrease of perfect margins was found only for SC and VLV. After TCML, SC and VU revealed a significantly better marginal adaptation than VLV. VU showed significantly fewer marginal imperfections than the other luting systems. After TCML, there was no significant difference in marginal gaps between the cervical and palatoincisal tooth/composite resin cement interfaces (Table 2). Furthermore, no significant differences could be found between the two cervical interfaces (dentin/composite resin cement and ceramic/composite resin cement; data not shown).

Cervical Ceramic/Composite Resin Cement Interface (Figure 3)

Before TCML, SC showed significantly fewer gaps than all other luting systems, while VU was

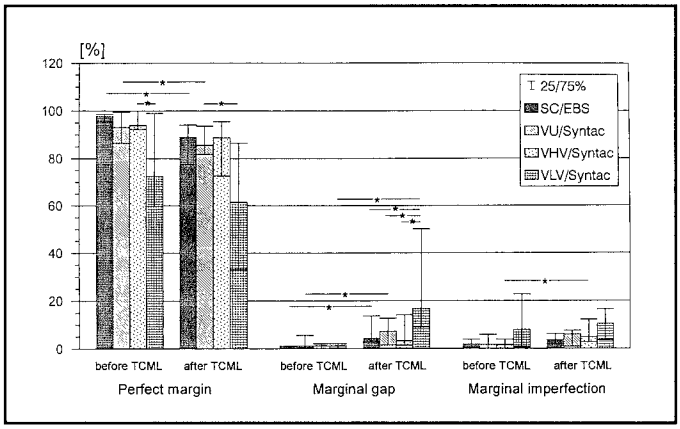

Figure 4. *Quantitative SEM margin analysis of the enamel/composite resin cement interface at the palatoincisal margin: median (25/75%) percentage of perfect margin, marginal gap, and marginal imperfection before and after TCML. Significant differences (P ≤ 0.05) between medians are labelled with *. SC = Sono-Cem; EBS = ESPE Bonding System; VU = Variolink Ultra; VHV = Variolink High Viscosity; VLV = Variolink Low Viscosity.*

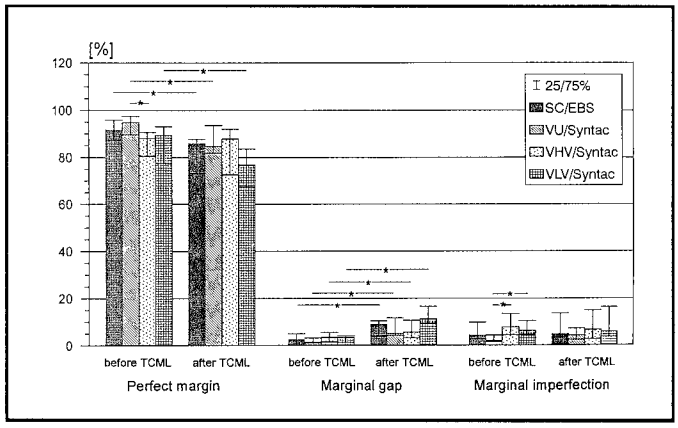

Figure 5. *Quantitative SEM margin analysis of the ceramic/composite resin cement interface at the palatoincisal margin: median (25/75%) percentage of perfect margin, marginal gap, and marginal imperfection before and after TCML. Significant differences (P ≤ 0.05) between medians are labelled with *. SC = Sono-Cem; EBS = ESPE Bonding System; VU = Variolink Ultra; VHV = Variolink High Viscosity; VLV = Variolink Low Viscosity.*

Table 2. Quantitative SEM Margin Analysis: Statistical Comparison of the Corresponding Interfaces at the Cervical and Palatoincisal Margins before and after TCML

			Sono-Cem	Variolink Ultra	Variolink High Viscosity	Variolink Low Viscosity
Dentin/Composite		perfect margin	*	ns	*	ns
at the Cervical Margin	before TCML	marginal gap	ns	ns	*	ns
vs		marginal imperfection	ns	*	*	ns
Enamel/Composite		perfect margin	*	ns	*	ns
at the Palatoincisal	after TCML	marginal gap	ns	ns	ns	ns
Margin		marginal imperfection	*	ns	*	ns
Ceramic/Composite		perfect margin	ns	ns	ns	ns
at the Cervical Margin	before TCML	marginal gap	ns	ns	*	ns
vs		marginal imperfection	ns	ns	ns	ns
Ceramic/Composite		perfect margin	ns	ns	*	*
at the Palatoincisal	after TCML	marginal gap	ns	ns	*	ns
Margin		marginal imperfection	ns	ns	ns	ns

* = statistically significant difference ($P \leq 0.05$); ns = no statistically significant difference.

significantly better than VHV. Both SC and VU showed significantly more perfect margins and fewer marginal imperfection than VHV and VLV. The marginal qualities were significantly worsened by TCML after using SC, VHV, and VLV, but not after using VU. After TCML, VU demonstrated significantly fewer marginal gaps than VHV and VLV, while all other luting systems revealed no significant differences. Comparing the cervical and palatoincisal ceramic/composite resin cement interfaces after TCML, only VHV showed significantly more marginal gaps at the cervical margin (Table 2).

Palatoincisal Enamel/Composite Resin Cement Interface (Figure 4)

Before TCML, there were no significant differences in the percentages of marginal gaps among the four luting sytems. However, after TCML, VLV showed a significantly higher percentage of marginal gaps than SC, VU, and VHV. VU was the only luting system, which was not significantly influenced by TCML. Furthermore, no significant differences could be found between the two palatoincisal interfaces (enamel/composite resin cement and ceramic/composite resin cement; data not shown).

Palatoincisal Ceramic/Composite Resin Cement Interface (Figure 5)

There were no significant differences in marginal gaps among the four luting systems before or after TCML. All luting systems revealed a significantly higher percentage of marginal gaps after TCML.

Microleakage (Dye Penetration)

The results and statistical analysis of the dye penetration are reported in Figures 6 and 7. At the cervical margin, which was located in dentin, SC revealed a trend towards a higher median percentage of dye penetration than the other luting systems, although the differences were not statistically significant (Figure 6). At the palatoincisal margin, which was located in enamel, in nearly all cases the penetration stopped before reaching the first dentinoenamel interface (grade 1; Figure 7).

DISCUSSION

Discussion of the Methods and Materials

The forces that are caused by thermal stress and polymerization shrinkage of the composite resin cements, as well as the low bond strength between composite resin cements and dentin, have been regarded as primary reasons for marginal failure of ceramic veneers in dentin (Sim & others, 1994; Tjan & others, 1989; Zaimoglu & others, 1992). The marginal adaptation of ceramic veneers to dentin might be improved either by increasing the bond strength, using more effective dentin bonding systems, or by reducing the polymerization shrinkage using composite resin cements of higher viscosity (Eick & others, 1997; Noack & others, 1992; Perdigao & others, 1994; Peutzfeldt, 1994; Sorensen & Munksgaard, 1996a,b; Walmsley & Lumley, 1995). In the present study, four dual-curing composite resin cements of different viscosities were applied. Beside a low-viscosity

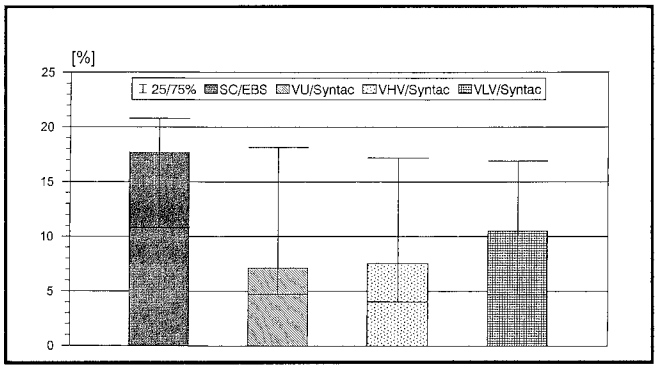

Figure 6. *Dye penetration at the cervical margin: medians (25/75%) of the maximum dye penetration (in % of the entire length of the cervical dentin involved in the restoration) after TCML. SC = Sono-Cem; EBS = ESPE Bonding System; VU = Variolink Ultra; VHV = Variolink High Viscosity; VLV = Variolink Low Viscosity.*

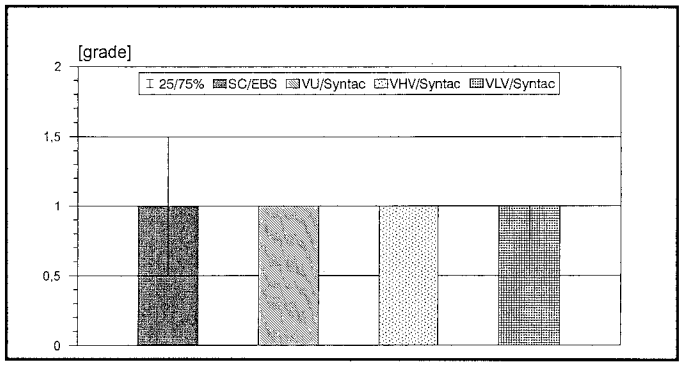

Figure 7. *Dye penetration at the palatoincisal margin: medians (25/75%) of the maximum dye penetration grades (0-3) after TCML. SC = Sono-Cem; EBS = ESPE Bonding System; VU = Variolink Ultra; VHV = Variolink High Viscosity; VLV = Variolink Low Viscosity.*

composite resin cement (VLV), which is routinely recommended for veneer restorations, composite resin cements (VHV, VU, SC) with a higher filler content were used. Two of the latter had to be inserted by the ultrasonic insertion technique (VU, SC). The thixotropic effect of vibrational energy facilitated good flow properties of these highly filled composite resins (Noack & others, 1992; Peutzfeldt, 1994; Walmsley & Lumley, 1995). Theoretically, highly filled composite resin cements show less polymerization shrinkage and better wear characteristics, preventing marginal ditching (Peutzfeldt, 1994), which might be of importance, especially for the palatoincisal margin of veneers.

In the present study, a high-strength, leucite-reinforced heat-pressed glass-ceramic was used for the fabrication of the veneers. The approximately 0.5 mm-thick veneers tolerated the application of the ultrasonic insertion technique without any damage. Similar to the procedure described by Zaimoglu and others (1992), the incisal edge was slightly reduced and the veneer was extended about 1 mm onto the palatal surface (Figure 1). Under clinical conditions, this would provide a better possibility to modify tooth morphology, a more natural translucency of the ceramic, a greater control of the anterior incisal guidance, and a reduction of the shear forces on the restoration (Harley & Ibbetson, 1991; Sheets & Taniguchi, 1990).

In contrast to previous in vitro studies on ceramic veneers, which used only dye penetration for the assessment of marginal adaptation (Sim & others, 1994; Tjan & others, 1989; Zaimoglu & others, 1992), quantitative SEM analysis and dye penetration were used in the present study. Within the limitations of in vitro studies, quantitative margin analysis by SEM has proven to be an exact and reliable method for evaluating marginal qualities of dental restorations (Peutzfeldt, 1994). In addition, dye penetration was used for the assessment of the marginal microleakage after TCML. While in previous studies (Sim & others, 1994; Tjan & others, 1989; Zaimoglu & others, 1992) the teeth were cut only once, in this study the evaluation of multiple sections of each tooth provided more detailed spatial information about the actual microleakage at the cervical and palatoincisal margins (Peutzfeldt, 1994). Since a restoration is only as good as its worst site, the maximum dye penetration of each tooth was used for further statistical evaluation. The main interest of this study involved the cervical veneer margin located in dentin. For this reason, the length of cervical dye penetration was expressed as percentage of the entire length of the dentin involved in the restoration, taking into account the different tooth sizes. At the palatoincisal margin, a semiquantitative evaluation method appeared to be more useful because of the alternating enamel and dentin interfaces. Due to the different assessment criteria, the dye penetration at the cervical and palatoincisal margins were not directly comparable. A further difference to previous studies (Sim & others, 1994; Tjan & others, 1989; Zaimoglu & others, 1992) was that, besides thermocycling, the ceramic veneers were subjected to incisal mechanical loading for a better clinical simulation. However, while in vivo loading of incisors is mostly at an angle, in the present in vitro study a simplified mechanical loading was used. The TCML machine required mechanical loading in an axial direction on the incisal edge of the veneers to facilitate an even load distribution. For this reason, load transfer was mainly in a compressive mode at the cervical and palatoincisal veneer/tooth interfaces, while load application at an angle might have caused tooth flexure. According to Krejci and Lutz (1990), 250,000 mechanical loading cycles in vitro correspond to the average mechanical loading in vivo during 1 year. For this reason, our protocol used 500,000 cycles to resemble in vivo mechanical loading during 2 years.

In this study, a positive control group of veneers with cervical margins located in enamel was omitted. Since a previous study (Sim & others, 1994) could not find any difference between cervical and incisal veneer margins, which were located in enamel, in the present study the palatoincisal enamel margin was used as positive control for the assessment of the integrity of the cervical veneer margins located in dentin. Axial mechanical loading on the incisal edge provided a similar load pattern for the cervical and palatoincisal veneer margins.

Discussion of the Results

Ceramic veneers, which were inserted using the highly viscous composite resin cements Sono-Cem (SC), Variolink Ultra (VU), or Variolink High Viscosity (VHV) with their corresponding dentin bonding agent, showed similarly favorable marginal adaptations to dentin and enamel. The quality of the marginal adaptation was similar at all four interfaces: the cervical dentin-composite resin cement and ceramic-composite resin cement interface as well as the palatoincisal enamel-composite resin cement and ceramic-composite resin cement interface. This is in contrast to previous in vitro studies (Sim & others, 1994; Tjan & others, 1989; Zaimoglu & others, 1992) of ceramic veneers with cervical margins located in dentin. However, a comparison is difficult, because those studies used only dye penetration for the assessment of the marginal integrity. In those studies, the dentin-composite resin cement interface showed statistically significantly more microleakage than the other three interfaces. While Tjan and others (1989) and Zaimoglu and others (1992) used no dentin bonding agents, Sim and others (1994) applied dentin bonding agents of an earlier generation. In all those investigations, the shrinkage forces of the composite resin cement resulted in a higher percentage of marginal gap formation compared to the enamel/composite or the silanated ceramic/composite interface.

In the present study, the bond strength between dentin and composite resin cement appeared to be as intact as the bond strength between enamel and composite or silanated ceramic and composite, which are both known to be predictable (Calamia, 1985; Simonsen & Calamia, 1983; Tjan & others, 1989).

At the cervical margin SC and VU exhibited a higher percentage of perfect margins and less marginal gap formation than VHV, but these differences were not statistically significant. However, VLV revealed a statistically significantly higher percentage of marginal gaps than the three highly viscous composite resin cements before and after TCML. The fact that VHV, VLV, and VU were used with the same dentin bonding agent (Syntac), as well as the superiority of VU and VHV over VLV, and the tendency for less gap formation with VU compared to VHV underlined the importance of a high filler content to reduce shrinkage forces. These results confirmed earlier findings on ceramic inlays with margins located in dentin or enamel showing a superiority of highly filled composite resin cements (Schmalz & others, 1995; Thonemann & others, 1994). In the present study, TCML caused a statistically significant increase in the percentage of marginal gaps for all composite resin cements at all four interfaces, with the exception of VU at the cervical ceramic-composite resin cement and the palatoincisal enamel-composite resin cement interface.

The dye penetration data at the cervical margin (Figure 6) confirmed the results, i e, relatively low percentage of marginal gaps and high percentage of perfect margins, which were found by quantitative SEM analysis. Assuming a maximum length of 2 mm (extension of preparation below the CEJ) for the cervical dentin involved in the restoration, a median dye penetration of about 7% corresponds to 0.14 mm. This is distinctly less than data reported in previous studies (0.5-4.3 mm) with and without using dentin bonding agents (Sim & others, 1994; Tjan & others, 1989; Zaimoglu & others, 1992).

This study confirmed results of previous in vitro and in vivo studies on veneers with margins located in enamel (Castelnuovo & others, 1996; Coyne & Wilson, 1994; Zaimoglu & others, 1992) that even under optimal conditions no treatment method so far is able to prevent marginal gap formation and microleakage completely in enamel or in dentin. However, these data indicate that ceramic veneers can be extended to cervical dentin without significant loss of marginal integrity compared to all-enamel preparations, provided that highly viscous composite resin cements and dentin bonding agents of the latest generation are used. Although SC and VU in combination with the ultrasonic insertion technique seemed to provide the best results, VHV facilitated similarly favorable marginal adaptation. This should be considered if ceramic systems are used that might be too brittle to allow veneer insertion by ultrasonic vibration.

CONCLUSIONS

Within the limitations of an in vitro study, the present data demonstrated that similarly favorable marginal adaptations of heat-pressed glass-ceramic veneers to dentin and enamel can be achieved using Sono-Cem, Variolink Ultra, or Variolink High Viscosity with their corresponding dentin bonding systems. Thermomechanical loading had a statistically significant influence on marginal gap formation at the cervical as well as at the palatoincisal margins and should be used for this kind of in vitro testing.

Acknowledgments

The authors thank Dr Karl-Anton Hiller for the statistical evaluation of the experimental data. We are grateful to Brigitte Bey for her help with the scanning electron microscope procedure. Furthermore, we want to thank Prof Dr John M Powers (University of Texas, Houston, TX) for reviewing this manuscript.

(Received 15 December 1997)

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Dentin Bond Strength and Marginal Adaptation: Direct Composite Resins vs Ceramic Inlays

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Clinical Relevance
Precuring the adhesive resin proved to be an important factor in direct composite restorations, but is not necessary for luting ceramic inlays. Recent one-bottle adhesives performed poorly compared with multi-step systems.

SUMMARY

The aim of this in vitro study was to compare the dentin bond strength and marginal adaptation of directly and indirectly inserted restorations. A conically modified push-out test was designed to consider polymerization shrinkage and facilitate inlay placement. A total of 260 cavities were prepared into disks of freshly extracted human third molars and filled with direct composite resins or with adhesively luted ceramic inlays. Dentin adhesives of the third- (with self-etching primer: ART Bond, Syntac Classic), fourth- (with total etching: Scotchbond Multi-Purpose Plus), and fifth-generation (one-bottle adhesives: Syntac Single Component, Prime&Bond 2.1) were used in combination with one hybrid composite (Tetric) or luting composite (Variolink Low). Control groups did not use an adhesive. Polymerization of the bonding agent was carried out prior to insertion of the filling/inlay or afterwards simultaneously with the composite/luting composite. The thickness of the adhesive layer and luting composite was recorded, and after 7 days of storage and 24 hours of thermocycling (1150 cycles) replicas were made and extrusion testing performed. Fracture modes were determined and replicas were examined regarding marginal adaptation using SEM (X200 magnification).

Precuring of the bonding resin increased dentin bond strength independent of the material combination or insertion mode ($P < 0.05$). In general, third- and fourth-generation dentin adhesives produced better results in bond strength and marginal adaptation than one-bottle systems ($P < 0.05$). In the third generation, ART Bond

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achieved significantly higher push-out values than Syntac ($P < 0.05$), but no better marginal adaptation. Cohesive fractures within the dentin were only observed in the inlay groups with precured resin.

Precuring of the bonding resin is an important factor for both direct and indirect restorations. Nevertheless, precuring of the bonding resin prior to insertion of adhesive inlays cannot be recommended clinically, because the 120-µm luting spaces were too large. In simulated cavities, direct composite fillings with precuring achieved bond strengths similar to inlays without precuring. One-bottle adhesive systems performed poorly compared with multi-step adhesives of the third and fourth generation.

INTRODUCTION

A stable and durable bond between dental materials and tooth substrates is important from a mechanical and esthetic standpoint (Walshaw & McComb, 1996). Good marginal adaptation prevents microleakage, recurrent caries, and pulpal irritation (Swift, Perdigao & Heymann, 1995). Acid etching of enamel produces an irregular surface that is perfect for bonding of unfilled resins (Buonocore, 1955). The result is a clinically successful retention and marginal seal of direct and indirect restorations, brackets, and pit and fissure sealants (Swift & others, 1995). Since the invention of the acid-etch technique, dentin bonding became one of the greatest challenges in restorative dentistry (Eick & others, 1991). Compared with the well-suited bonding to enamel, dentin is a less favorable substrate because of the high organic content, the smear layer, the tubular structure, the variation in the degree of mineralization, and the presence of outward fluid movement in the dentinal tubules (Walshaw & McComb, 1995; Eliades, 1994; Nakabayashi, Ashizawa & Nakamura, 1992). With encouraging results since the early 1990s, the development of new dentin adhesive systems has progressed very quickly (Watanabe & Nakabayashi, 1994). Recent dentin adhesive systems became more and more clinically acceptable by improving the adhesion to dentin in several steps with separate components for priming and bonding (Triolo, Swift & Barkmeier, 1995). Reports of the fifth generation of dentin adhesive systems and the process of development continue (Mason, Calabrese & Craif, 1997; Kanca, 1997; Finger & Fritz, 1996). The so-called one-bottle dentin adhesive systems were developed, promising good and effective bonding to

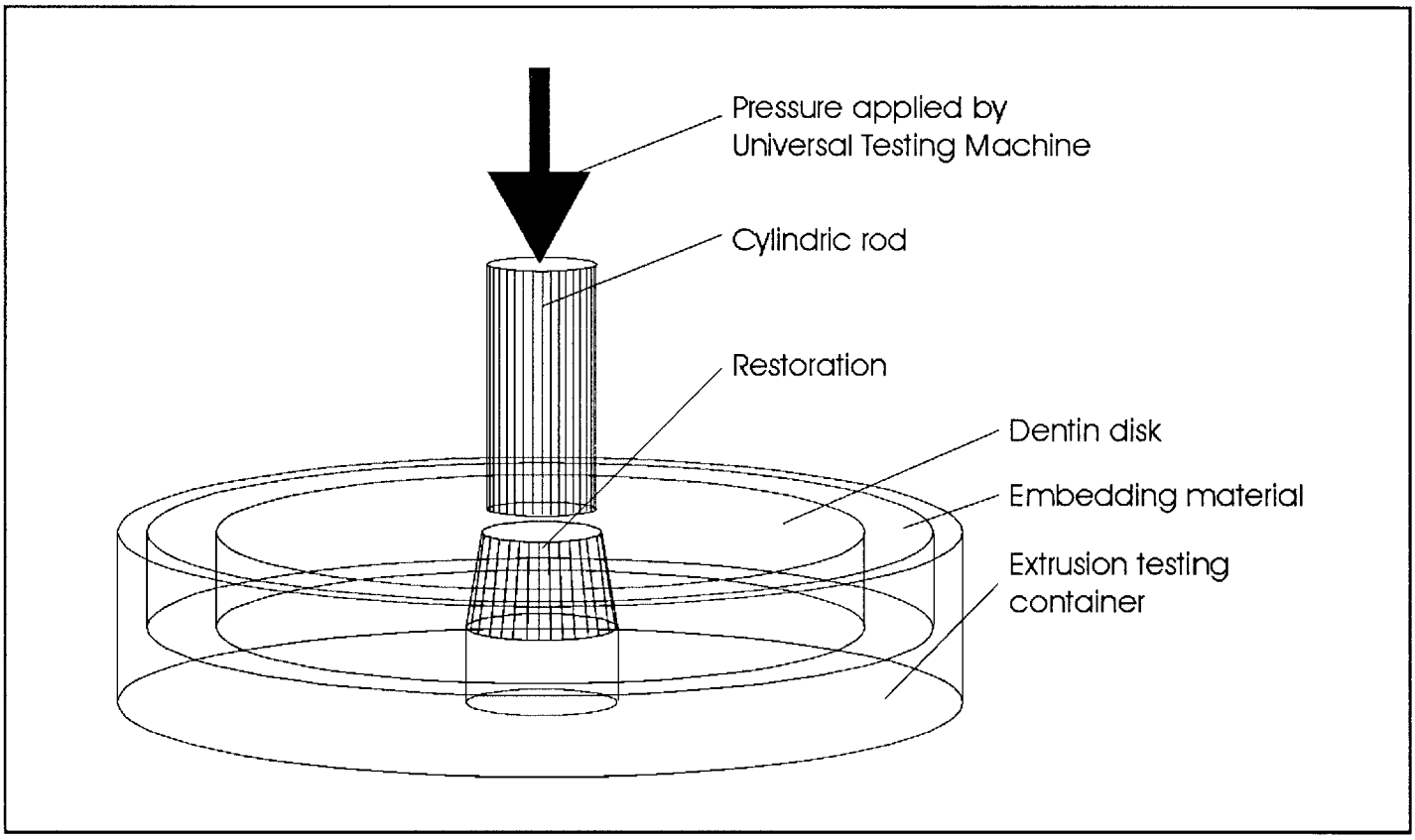

Figure 1. *Conic extrusion device*

Bonding Procedures, Batch Numbers, Dentin Pretreatment, and Manufacturers of the Products Tested

Bonding System	Bonding Steps	Batch #	Dentin Pretreatment	Manufacturer
ART Bond (AB)	Primer A, Primer B, Bond	9805FE879 9805FE879 9805FE879	Apply mixture of primers for 30 seconds, dry. Apply bond, air thin. Light cure for 20 seconds.	Coltène/Whaledent, Alstätten, Switzerland
Syntac (SY)	Primer, Adhesive, Bond	614334 614377 611589	Apply primer for 30 seconds, dry. Apply adhesive for 30 seconds, dry. Apply bond, air thin. Light cure for 20 seconds.	Vivadent, Schaan, Liechtenstein
Scotchbond Multi-Purpose Plus (SB)	Etchant, Primer, Adhesive	3 DK 3 CL 3 CB	Apply etchant for 15 seconds, rinse and dry. Apply primer, air thin. Apply adhesive, air thin and light cure for 10 seconds.	3M Dental Products, St Paul, MN 55144
Syntac Single Component (SC)	Etchant, Single Component Adhesive	625987 702027	Apply etchant for 15 seconds, rinse and dry. Apply SC-adhesive for 30 seconds, air thin and light cure for 10 seconds. Apply second coat, air thin and light cure for 10 seconds.	Vivadent
Prime&Bond 2.1 (PB)	Etchant, Primer/Bond	950148 950366	Apply etchant for 15 seconds, rinse and dry. Apply SC-adhesive for 30 seconds, air thin and light cure for 10 seconds. Apply second coat, air thin and light cure for 10 seconds.	DeTrey/Dentsply, Konstanz, Germany

dentin with a single adhesive solution for priming and bonding (Mason & others, 1997; Kanca, 1997). These materials consist of hydrophilic and hydrophobic resins simultaneously dissolved in solvents like alcohol or acetone, displacing water, and achieving an intimate contact to dentinal structures (Finger & Fritz, 1996). These dentin adhesive systems also require several treatment steps because of multiple coating, but nevertheless the very easy handling conditions make them very popular with general dental practitioners.

Dentin bonding, however, is not confined to direct composite restorations. Tooth-colored inlays are being seated more and more often by using dentin adhesive systems due to their successful prevention of postoperative hypersensitivity (Hickel, 1990; Kelsey & others, 1996; Kielbassa & others, 1996).

The selected testing design for the present investigation was a conic extrusion model (Frankenberger & others, 1997; Figure 1). Extrusion testing in adhesive dentistry, first described by Roydhouse (1970) and Kimura (1985), involves pushing out composite cylinders from dentin disks (Watanabe & Nakabayashi, 1994). Haller and others (1991) presented the first results of more recent adhesive sytems. Recently, the push-out design was taken up again for evaluating bond strength of composite resins applied in vivo (Mason & others, 1997). A further push-out study was conducted in internal dentin for endodontic reasons (Patierno & others, 1996).

It was the purpose of this in vitro study to evaluate the dentin adhesion behavior of direct and indirect tooth-colored restorations in combination with dentin adhesive systems of the third, fourth, and fifth generations. The study was carried out by determination of dentin push-out bond strength and marginal adaptation. Particular attention was given to the moment of adhesive resin polymerization.

METHODS AND MATERIALS

Specimen Preparation and Bonding Procedures

A total of 260 caries-free human third molars, stored in 0.1% thymol solution at ambient temperature

for less than 4 weeks after extraction, were used in this investigation. The teeth were debrided and examined to ensure that they were free of defects. Disks of 2 mm in diameter were cut from the mid-coronal level of the tooth, perpendicular to the tooth axis. One central conic cavity was prepared into each disk using standardized conic burs (Cerafil bur, Komet Inc, Lemgo, Germany). After preparation, the dentin disks were embedded in a temporary restorative material (Provipont, Vivadent, Schaan, Liechtenstein). The specimens were randomly assigned to 26 groups (n = 10).

Direct Composite Resin

Sixty disks were filled with one direct composite resin (Tetric, Vivadent, batch-number 618465, shade A 2). The cavity surfaces were treated with dentin bonding agents of different generations according to the manufacturers' instructions. The steps of dentin pretreatment are displayed in the table. The adhesive resin was polymerized prior to the application of the composite resin.

Sixty disks were treated identically, except for separate polymerization of the bonding resin. As a negative control, 10 disks were filled without using a dentin adhesive.

During the application of the composite resin, the specimens were placed on a glass sheet. The direct composite resin was inserted in one increment and condensed with a plugger. Excess composite was carefully removed with an explorer. The composite was cured for 60 seconds from each of two directions with contact to the composite surface using a matrix band (Frasaco strip, Franz Sachs & Co, Tettnang, Germany) as separating medium. Bonding agent and composite were cured with an Elipar II curing light (ESPE, Seefeld/Oberbay, Germany). The intensity of the light was checked periodically with a radiometer (Demetron Research Corp, Danbury, CT 06810) to ensure that 400 mW/cm^2 was exceeded.

Ceramic Inlay

The second half of the specimens were restored with prefabricated IPS-Empress ceramic inserts (Cerafil, Komet) for simulation of the indirect method. After preparation of the cavities, the cavity walls were treated identically like the composite resin groups, partly with and without precuring the bonding agent. In the one-bottle groups, two coats of prime/bonding agent were applied. Ten cavities were restored without using a dentin adhesive system. The inserts were etched with 5% hydrofluoric acid (Vita Ceramic Etch, Vita Zahnfabrik, Bad Säckingen, Germany) for 60 seconds and rinsed for 60 seconds. Consecutively, the silane coupling agent Monobond S (Vivadent) was applied and air dried. Then a bonding resin (Heliobond, Vivadent) was painted onto the silanated surface of the insert and air thinned. The disks were placed onto a glass sheet for simulating the cavity floor of a clinical situation. Afterwards, the cavity was completely filled with one luting composite (Variolink Low, Vivadent, batch numbers: base 614053, catalyst 616853) and the insert was placed. After seating of the inlay, the glass sheet was removed to ensure the final position of the inlay. After removal of excess luting resin composite with an explorer, the restorations were polymerized for 60 seconds from both sides, as it was carried out in the composite groups. Each specimen was ground flat and polished with #600 SiC paper under running water perpendicular to the push-out direction. The filled disks were stored in distilled water for 7 days at 37 °C.

Thermal Loading

After storage the specimens were subjected to an alternating thermal cycle of +5 °C and + 55° C in a thermocycling apparatus for 24 hours (1150 cycles). The dwell time at each temperature was 30 seconds, and the transport time between the water baths was 15 seconds. Then replicas were produced for analyzing marginal adaptation.

Light Microscopic Evaluation

After impressions were taken with a polyvinyl siloxane material (Permagum, ESPE), epoxy replicas (Epoxy Die, Vivadent) were produced and then analyzed using a light microscope (Wild M3Z, CH-Heerbrugg, Switzerland). The largest film thickness of the adhesive resin (directly restored cavities) and the luting space (inlay groups) was measured for each specimen at X40 magnification using the image analyzing system TiffMes 1.9 (University of Erlangen, Germany).

Push-out Testing

Finally, the specimens were positioned into the extrusion device (Figure 1) and mounted in a Universal Testing Machine (Zwick Corp,Ulm, Germany). A cylinder-shaped rod (1.7 mm in diameter) was attached to a compression load cell and, traveling at a crosshead speed of 0.5 mm/min, was applied to each filling or inlay until failure occurred. Failure was defined as the 30% loss of the maximal push-out force. The push-out bond strength was determined by computing the quotient of maximum load (N) and adhesion area (truncated cone; mm^2).

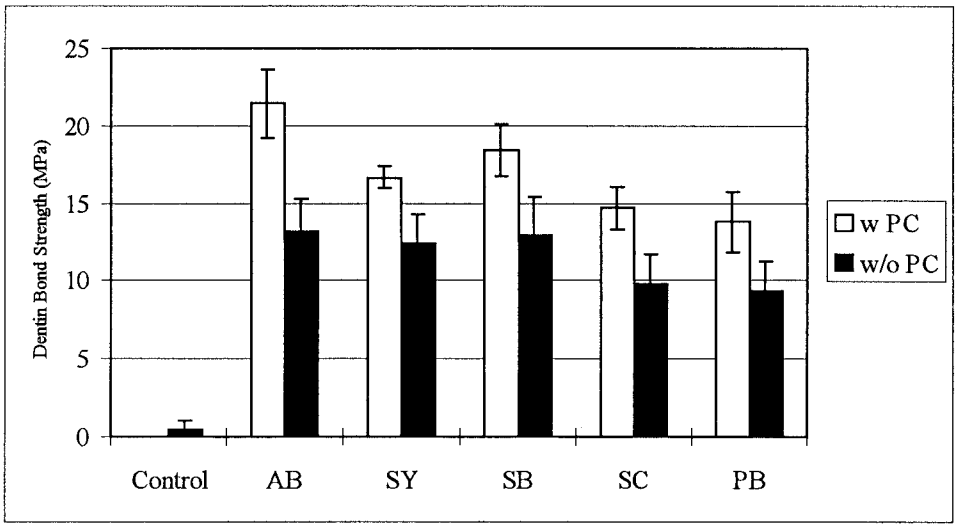

Figure 2. *Mean push-out bond strength (MPa) of the direct composite resin groups with control (no adhesive tested). w PC = precured adhesive resin; w/o PC = without precured adhesive resin. Vertical bars indicate standard deviation.*

Scanning Electron Microscopic (SEM) Evaluation

The replicas were sputter-coated with gold (Sputter device: Balzers SCD 40, Balzers, Vaduz, Liechtenstein) and the interfaces analyzed under a scanning electron microscope (Leitz ISI 50, Akashi, Tokyo, Japan) at a magnification of X200. A quantitative analysis of the margins according to the criteria "gap-free margin" or "gap/irregularity" was performed using the image analyzing system TiffMes 1.9. Marginal quality was calculated as a percentage of gap-free margins related to the entire length of the particular margin. Marginal gaps and marginal irregularities were not recorded separately.

After the push-out procedure, the original specimens were mounted on aluminum cylinders, sputter-coated, and observed by SEM at X100 magnification to determine the fracture modes of the extruded restorations.

Statistical Analysis

The statistical analysis was performed using SPSS/PC+, Vers 7.5, SPSS inc, Chicago, IL 60611) for Windows 95/V 7.5. The values of push-out bond strength and marginal adaptation were non-normally distributed (proved by Kolmogorov-Smirnov test); therefore, a nonparametric test (Mann-Whitney-U test) for pairwise comparisons at the 0.05 level of significance (α) was performed. The data of the adhesive layer and luting space thickness were normally distributed; therefore, the mod LSD test (ANOVA) at the 0.05 level of significance was utilized. The levels of significance were adjusted to $\alpha^* = 1-(1-\alpha)^{1/k}$ (k = number of performed pairwise tests) to assess the influence of the different materials.

RESULTS

Composite Resin Filling

The mean push-out bond strengths for the composite restorations are displayed in Figure 2. Without dentin adhesive, the average push-out bond strength was 2.9 MPa. Without separate polymerization of the bonding resin, no significant differences between the testing groups occurred. The values were significantly lower than all other groups tested in this study, and resulted in the lowest level of significance. After precuring the resin, the push-out bond strength increased significantly in all groups, and significant differences between the groups could be observed. AB resulted in statistically higher push-out bond strength than the other material combinations; the next level is represented by SB and SY. SC and PB showed the lowest push-out bond strength ($P < 0.05$; Mann-Whitney-U test). Adhesive film thicknesses ranged between 5 and 65 µm. The one-bottle adhesive systems revealed significantly

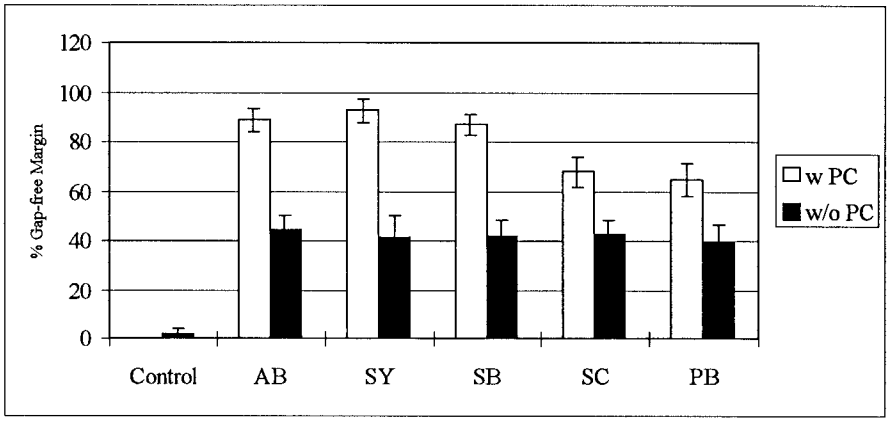

Figure 3. *Percentages of gap-free margins at the dentin-composite interface in the composite resin groups. w PC = with precured adhesive resin; w/o PC = without precured adhesive resin. Vertical bars indicate standard deviation.*

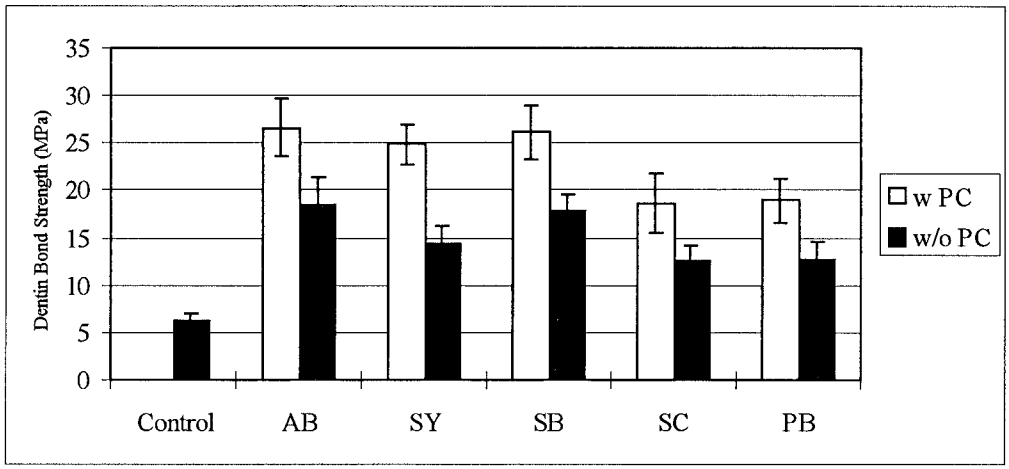

Figure 4. *Mean push-out bond strength (MPa) of the inlay groups with control (no adhesive used). w PC = with precured adhesive resin; w/o PC = without precured adhesive resin. Vertical bars indicate standard deviations.*

thicker adhesive layers than the multi-step systems ($P < 0.05$; mod LSD-test/ANOVA).

The means of gap-free margins in the directly restored groups are displayed in Figure 3. The percentages of gap-free margins were significantly higher using adhesive systems of the third and fourth generation (SY 92.5 %, AB 88.6 %, SB 86.7 %) in comparison to the single-bottle dentin adhesive systems (SC 67.8 %, PB 64.6 %). Without precuring the bonding resin the percentages of gap-free margins decreased to <50% in all groups. The observed fracture mode was mostly adhesive.

Ceramic Inlay Technique

The mean push-out bond strengths for the adhesive inlay formations are listed in Figure 4. The bond strength without precuring the resin was significantly lower ($P < 0.05$), as it was observed in the direct groups. Analogously to the direct groups the time-expensive multi-step systems produced better push-out bond strength results than the one-bottle systems ($P < 0.05$). Using AB, SY, and SB, the precured samples revealed more than 98% gap-free margins after storage and thermocycling (Figure 5). After curing the resin, the push-out bond strengths were significantly higher than without ($P < 0.05$). However, if multi-step systems were used without precuring, the part of gap-free margins was also more than 90%. The precured one-bottle groups showed significantly higher percentages of gap-free margins and bond strengths than without curing of the bonding resin (Figure 5). The push-out bond strength without adhesive system was 6.2 MPa. Luting composite film thicknesses of the precuring groups were significantly greater than without curing ($P < 0.05$, mod LSD test/ANOVA). The widths of the maximum luting space ranged from 44 µm to 112 µm. The fifth-generation adhesives revealed significantly greater luting spaces than the multi-step systems of the previous generations ($P < 0.05$). The observed fracture modes were adhesive in the groups without precuring. In the groups with precuring of the resin, up to 60% cohesive fractures occurred with completely crushed dentin disks.

The push-out bond strength of directly applied composite restorations with prepolymerization of the bonding agent showed similar results to the inlay groups without separate light-curing of the adhesive.

DISCUSSION

The importance of precuring the adhesive resin for direct composite restorations is reported in some studies (McCabe & Rusby, 1994; Paul & Schärer, 1997). Nevertheless, according to easy handling

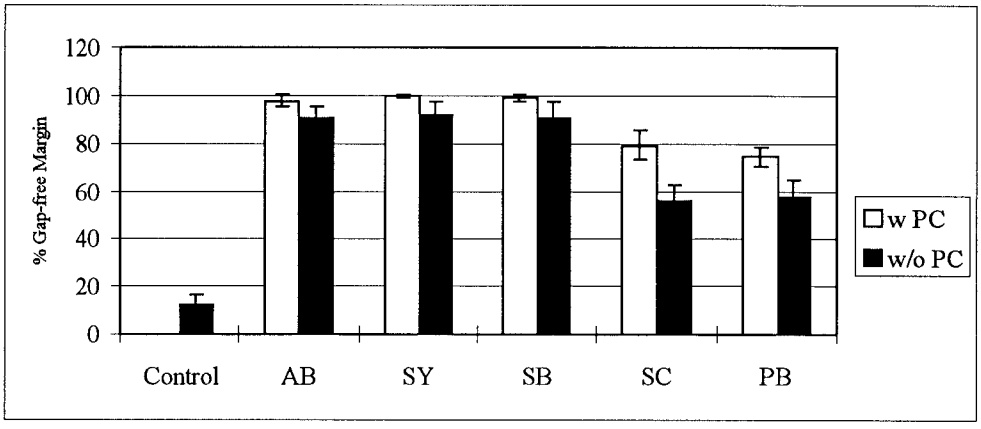

Figure 5. *Percentages of gap-free margins at the dentin-luting composite interface in the inlay groups. w PC = with precured adhesive resin; w/o PC = without precured adhesive resin. Vertical bars indicate standard deviation.*

conditions, the manufacturers tend to omit this step in adhesive dentistry. This study should clarify this problem using a simulated cavity design for simultaneously testing bond strength and marginal adaptation of the restorations. The push-out design was thus selected. It allowed both bond strength testing and margin analysis in one specimen (Haller & others, 1991). The extrusion design simulated polymerization stresses as they were found in cavities and revealed a configuration factor of about 1.7 for direct restorations and 20 for indirect filling techniques compared to a factor of about 0.2 in shear or tensile procedures using bonded composite cylinders (Feilzer, de Gee & Davidson, 1987). A configuration factor is defined as the relationship between a bonded and nonbonded composite surface. An interesting point of the present study was the obvious difference in the volume of the composite resin and luting composite. Only extrusion testing can consider different polymerization stresses similar to clinical practice. Furthermore, in extrusion testing no statistically significant difference was found between in vitro and in vivo testing (Mason & others, 1997).

A conic modification of Haller's design was developed to enable evaluation of inlay placement in push-out testing. The 4° cone of the Cerafil set was a standardized device for this modification. The self-centering effect of the conic model allowed the insertion of prefabricated inlays.

Due to the negative influence of hygroscopic expansion of various filling materials for the use of direct and indirect restorations, *one* composite resin and *one* luting composite were utilized to exclude changes in volume or stress distributions of different composite materials (Rees & Jacobsen, 1992).

In the inlay groups, no temporary material and no provisional cement were used, in order to compare the groups without interfering influences of these materials (Paul & Schärer, 1997; Rotberg & deShazer, 1966; Xie, Powers & McGuckin,1993).

The results showed that the newer single-bottle dentin adhesive systems performed worse in bonding direct composite resins to dentin than multi-step adhesives. Analogously, for one-bottle adhesives shear bond strength values are reported to be in the range of 2.9 - 12.3 MPa (Swift & others, 1997; Vargas, Fortin & Meckes, 1995). This poor performance might be related to inadequate resin penetration into the dentin surface and seemed to be independent from the agent used for dissolving the hydrophilic resins. Neither Prime&Bond 2.1 (acetone) nor Syntac Single Component (water) achieved an encouraging push-out bond strength. Nevertheless, high bond strengths to dentin depend not only on good penetration into the demineralized surface, but also on the mechanical properties of the enamel/dentin adhesive resin (Finger, Inoue & Asmussen, 1994).

The additional marginal analysis of the present study also revealed poor marginal adaptation by using the tested single-bottle adhesive systems. Therefore, this was an indication that the results of the investigated one-component dentin adhesives were attributed to an unfavorable penetration into the dentin surface. The results might indicate that separate steps of dentin treatment improved the bonding efficacy to dentin.

Comparing direct fillings with precured and non-precured adhesive resin, the materials showed equal results concerning push-out bond strength and marginal adaptation. Using the precuring procedure, the multi-bottle dentin adhesive systems reported better results to push-out bond strength and marginal adaptation, but without precuring no significant differences were found. This showed that the precuring procedure has to be performed if direct composite restorations are used, as confirmed in the literature (Erickson, 1989; Crim, 1990; McCabe & Rusby, 1994).

The evaluation of the inlay technique showed different results. Without precuring of the bonding agent, the push-out bond strength result was comparable to directly applied composite resins. The performance of the single-bottle adhesives was not promising in comparison with multi-step systems. The marginal adaptation revealed results of more than 90% gap-free margins with the systems AB, SY, and SB, although no precuring of the resin was performed. This observation verified other in vitro studies (Schmalz, Federlin & Reich, 1995; Sorensen & Munksgaard, 1996). Nevertheless, using the precuring procedure, gap-free margins increased up to >98%, and the push-out bond strength values were significantly higher than without separate irradiation of the bonding agent. But it has to be taken into account that the widths of the luting spaces were more than 100 μm in the precured groups. Like in a cavity preparation, only a small area of insufficiently air-thinned adhesive resin transmitted to the luting space. These results suggest that by the use of this technique in clinical situations, the ceramic inlay cannot be placed into its final position, especially if pools of the translucent bonding resin are overlooked prior to its photopolymerization. Double coating of the resin was aggravating for the one-bottle systems and led to luting composite thicknesses of more than 100 μm, but the manufacturer's conditions had to be observed.

Therefore, the curing of the air-thinned adhesive resin prior to the application of the luting composite cannot be recommended for clinical practice. Besides, the push-out bond strength and marginal adaptation results without precuring of the bonding agent were also encouraging.

Nevertheless, the different elastic modulus of the

tested materials (composite 10 GPa vs ceramic 70 GPa) must be considered. This fact could be responsible for the cohesive dentin fractures in the precured inlay groups and could explain the comparatively high push-out bond strength in these groups.

Results showed that the tested one-bottle adhesive systems could not be recommended for luting procedures because the manufacturers advise a double coating and double curing of their single-bottle dentin adhesive systems, leading to thick layers. Composite resin results of marginal adaptation and push-out bond strength showed that clinical success could be expected with these materials if the dentin adhesive system was precured. Nevertheless, luting adhesive inlays by use of multi-step adhesive systems without curing the bonding agent prior to insertion of the luting composite shows promise in the clinical environment.

CONCLUSIONS

For best results using a direct restorative treatment, the adhesive resin must be light-cured prior to application of a resin composite material. The more time-expensive dentin adhesives produced higher bond strengths and better marginal adaptation in comparison to single-bottle adhesives. The indirect restoration of dentinal cavities showed an encouraging adhesion without precuring the adhesive. Light curing the resin prior to insertion of the ceramic inlay produced almost gap-free margins and high bond strengths, but large diameters of luting space could cause an insufficient fit of the inlay after placement or fracture of the inlay under high insertion pressure by the clinician. Therefore, single-bottle adhesives should be used with caution when luting tooth-colored inlays because of the large luting spaces involved and the poor push-out bond strength and marginal adaptation results obtained in this study.

(Received 20 January 1998)

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Shear Bond Strength of Repaired Composite Resins Using a Hybrid Composite Resin

C W SAU • G S Y OH
H KOH • C S CHEE • C C LIM

Clinical Relevance
The hybrid composite resin produced minimally adequate shear bond strength when it was used to repair the composite resins used in this study.

SUMMARY

The shear bond strength of different types of composite resins repaired with a hybrid composite was evaluated. The hybrid composite resin was repaired with itself as a control. The results of this study showed that the shear bond strength of the repair with most types of composite resins was minimally adequate.

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INTRODUCTION

Buonocore (1955) was the first to discuss the acid-etch technique for increasing the adhesion of acrylic filling material to enamel. Since then, research and development of composite resins have come a long way. The use of resin restorative materials has been extended from simple class 3 preparations to those of greater complexity and size.

Techniques for the mechanical preparation of fractured surfaces to enhance the retention of new composite resins to old composites have been widely evaluated and reported (Caspersen , 1977; Gwinnett & Matsui, 1967; Retief, 1978; Boyer & Hormati, 1980; Boyer, Chan &Torney, 1978). Chemical bonding has also been studied by Vankerckhoven and others (1982). They found that the residual unreacted methacrylate groups in microfilled and in large-particle composite resins after curing was 40% and 50% respectively. Therefore, chemical bonding of new composite resins to previously polymerized resins could be expected.

Several laboratory investigations (Boyer & others, 1978; Boyer, Chan & Reinhardt, 1984; Söderholm,

1985) have also demonstrated that surface preparations of old composite resins by etching with 30%-50% phosphoric acid and treating with unfilled bonding agents optimized the repair bond strength. A hybrid composite resin system (Z100) was chosen as the material for repair due to its popular usage clinically as compared to conventional and microfilled composite resins. It has been shown that hybrid composite resins possess comparable mechanical strength to conventional composites and are superior to microfilled resins (Phillips, 1991). The hybrid composite resins also have a polishability that is comparable to that of microfilled composite resins and better than that of macrofilled composite resins (Phillips, 1991).

The aim of this in vitro study was to evaluate the shear bond strength of four different types of composite resins repaired with a hybrid composite resin (Z100, 3M Dental Products, St Paul, MN 55144) up to 4 weeks. A control was established by repairing the hybrid composite resin with itself. The hybrid composite resin in this study is one with filler particles containing both conventional (1-5 microns) and microfill particles (0.04 microns) (Caspersen, 1977).

METHODS AND MATERIALS

The composite resins that were used in the study included a conventional composite resin, Epolite100 (GC America, Chicago, IL 60658); a polyacid-modified composite resin, Dyract (Dentsply, Weybridge, UK); a microfill composite resin, Silux Plus (3M Dental Products); a submicron hybrid composite resin, Spectrum (Dentsply); and a hybrid composite resin, Z100 (3M Dental Products). Epolite 100 is a chemical-cure composite resin, while the rest of the composite resins were light-cure resins. The resins used had different ranges of particle sizes (Table 1). All the materials were manipulated according to the manufacturers' instructions.

Cylindrical jigs 30 mm in diameter and 40 mm in height were fabricated from autopolymerizing Tray Resin II (Shofu Inc, Kyoto, Japan). Each jig had indentations of 9.2 mm in diameter and 1.5 mm in depth in the center on both ends. Four retention points were created at the line angle between the axial surface and the floor of the indentations by using a # 5 stainless steel inverted cone bur fixed onto a slow-speed handpiece (Figure 1).

Thirty samples of each type of composite resin were prepared by condensing them into the indentations with a clean plastic instrument. This was to ensure minimal contamination and incorporation of voids into the study materials. These materials, which were packed into the indentations, will be referred to as "parent materials." A glass slide was then placed over the surface of the packed material. For the light-cure materials, a light-curing tip (L D Caulk, The Maxlite, Model 103/240, Serial 20099, L D Caulk, Milford, DE 19963) was placed in contact with the glass slide over the material and cured for 40 seconds. Before curing each sample group, the intensity of the light emission from the light-curing unit was noted using Cure-Rite (EFOS Inc, Mississauga, Ontario, Canada). This was to ensure that the light intensity was more than the recommended 300 W/m^2 at any one time. The glass slide was removed 10 minutes later, and the samples were stored in a water bath of 37 °C for 24 hours.

After 24 hours, the samples were removed from the water bath. Every exposed surface of the parent material was prepared by passing a 600-grit Sof-Lex disk (3M Dental Products) for five strokes only in one direction with a low-speed handpiece at 1000 rpm. A 2-second movement of the disk across the diameter of the sample surface constituted a "stroke."

Debris left behind by the above preparations was washed off for 5 seconds and the samples dried using compressed air for 2 seconds. Scotchbond etchant 35% phosphoric acid (3M Dental Products) was applied to the sample surfaces for 15 seconds. The etchant was then washed off with water for 15 seconds and dried using compressed air for 2

Table 1. Materials Used

Brand Name	Description	Filler Particle Size (micrometers)	Filler Loading (% by volume)
Epolite	Chemical-cure conventional composite resin	unavailable	unavailable
Dyract	Light-cure compomer	Mean 2.5	72 (w/w)
Spectrum	Light-cure submicron hybrid composite resin	0.04 - 5	57
Silux Plus	Light-cure microfill composite resin	0.04 - 0.09	40
Z100	Light-cure hybrid composite resin	0.01 - 3.5	66

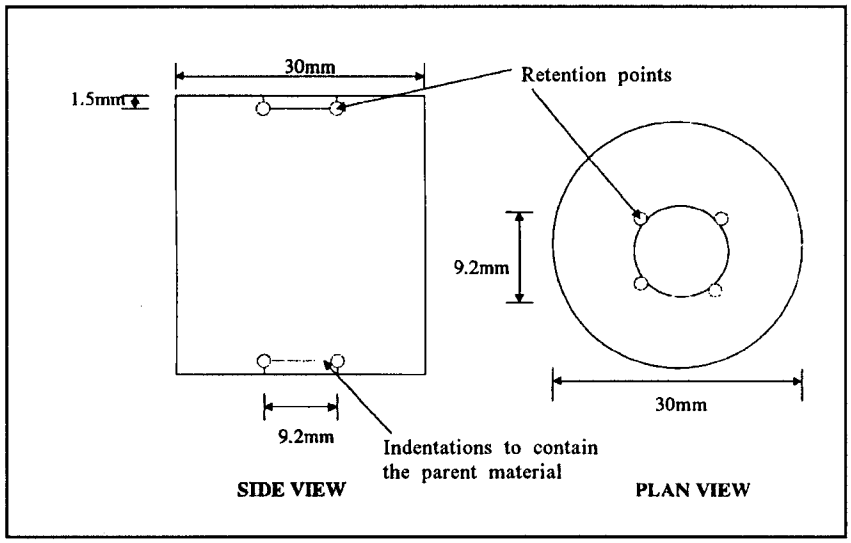

Figure 1. *Dimensions of cylindrical jig for composite resin samples*

seconds. Scotchbond Multi-Purpose primer (3M Dental Products) was applied to the etched sample surface and dried gently using compressed air for 5 seconds. A thin layer of Scotchbond Multi-Purpose adhesive (3M Dental Products) was then applied and cured for 10 seconds.

A plastic matrix of external diameter 3.73 mm and height 1.5mm (Figure 2) was placed over the prepared surface of each sample. The hybrid composite resin, Z100, was packed and condensed into the matrix and then light cured for 40 seconds. The Z100 placed in the above matrix together with the adhesive system used will be referred to as "repair material," while the parent material with the repair material bonded on will be referred to as the "repaired sample." Thirty repaired samples of each parent material were divided into three groups of 10.

Group 1

Ten repaired samples of each parent material were tested immediately after the repair material was cured with an Instron Universal Testing Machine, Model 4502 (Instron Corp, Canton, MA 02021) until bond failure occurred. The crosshead speed was set at 5 mm/min and load cell of 1 kN. The shear apparatus utilized a load cell applied at the line angle between the parent material and the repair material, which resulted in a 90° load application angle.

Group 2

Ten repaired samples of each parent material were stored in a water bath of 37 °C for 1 week and the shear bond strength tested using the same regime as in Group 1.

Group 3

Ten repaired samples of each parent material were stored in a water bath of 37 °C for 4 weeks and the shear bond strength tested using the same regime.

RESULTS

The immediate, 1-week, and 1-month shear bond strengths of the repaired samples are shown in Table 2 and Figure 3. The mean shear bond strength values immediately after repair ranged from 3.68 MPa to 10.27 MPa, with Spectrum having the greatest value. Epolite had the second highest shear bond strength, followed by Dyract, Z100, and then Silux Plus.

At 1 week, Silux Plus had the greatest shear bond strength, followed by Dyract, Epolite, Z100, and then Spectrum. The range of values was from 10.04 MPa to 18.50 MPa.

At 4 weeks the greatest mean shear bond strength was observed in Dyract. This was followed by Z100, Spectrum, Epolite, and then Silux Plus. The values ranged from 5.63 MPa to 8.13 MPa.

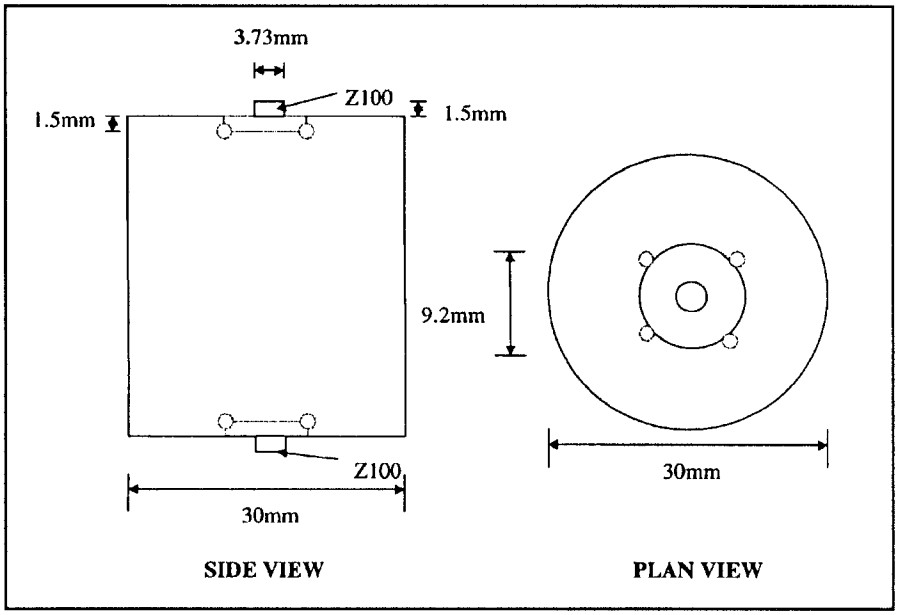

Figure 2. *Repaired samples prepared on the cylindrical jig*

Table 2. Mean Shear Bond Strengths of Repaired Materials

	Mean Shear Bond Strength in MPa (SD)		
Material/ Time	**Immediate**	**1 Week**	**4 Weeks**
Dyract	4.86 (1.99)	16.68 (9.55)	8.13 (1.90)
Silux Plus	3.68 (1.61)	18.50 (7.57)	5.63 (2.12)
Spectrum	10.27 (4.87)	10.04 (5.01)	7.04 (2.25)
Epolite	8.79 (4.42)	11.98 (6.06)	6.05 (1.42)
Z100	4.05 (0.81)	11.19 (5.72)	7.51 (3.33)

Using one-way ANOVA and Scheffé's test ($P < 0.05$), comparisons were made between the repaired materials at each time interval (Table 3). Comparisons were also made between the various time intervals for each material (Table 4).

At the time of immediate repair, Epolite showed significantly greater shear bond strength than Silux Plus and Z100. Spectrum had significantly greater shear bond strength than Silux Plus, Z100, and Dyract. However, no significant difference was noted in the shear bond strength between the parent and repair materials after 1 week and 4 weeks.

For Dyract and Silux Plus, the shear bond strength of the repair at the 1-week interval was significantly greater than the immediate and 4-week shear bond strength. For Spectrum no significant difference was observed in the shear bond strength at any of the time intervals. For Epolite, the only significant difference in shear bond strength noted was that of 1 week being more than 4 weeks. For Z100, the shear bond strength for 1 week was significantly greater than that immediately after repair. However, there was no significant difference between that at 1 week and 4 weeks after repair.

DISCUSSION

Repair in this context refers to the adding of fresh material to a material that already has been cured. In this study different types of composite resins were used as the parent material. This was to simulate clinical situations whereby patients may present with defective restorations requiring repair. The clinician may not be able to determine the type of restoration previously placed. Hence this study determined whether Z100 (3M Dental Products), a hybrid composite resin, provided adequate shear bond strength when used as a repair material on other types of composite resins. The different types of composite resins included light-cured and chemical-cured resins that are comprised of different resins containing various particle sizes.

Shear bond strength was tested because it provided the most appropriate measure of the maximum stress applied at the bonding interface between the parent material and the repair material. These restorations when used in the anterior region are commonly subjected to shear forces during mastication. Hence the study of shear bond strength of these repairs would be useful in predicting their success (Caspersen, 1977).

A comparison was done between the shear bond strength of repair using like materials, in this case Z100 to Z100 (control), and using unlike materials: Z100 to the other parent materials. At the time of shear immediately after repair, Spectrum and Epolite showed significantly higher shear bond strength to Z100. The bond strength of a repair depends on the amount of copolymerization between the unreacted methacrylate groups in the parent material with the adhesive. In addition, the rate of copolymerization determines the repair bond strength at a stipulated time. The faster initial rate of formation of cross-linkages between the parent materials of Spectrum and Epolite could account for the high immediate shear bond strength of the repair. The rate of formation of cross-linkages could progress at a slower rate in the repair of Dyract, Silux Plus, and Z100, resulting in a lower immediate shear bond strength.

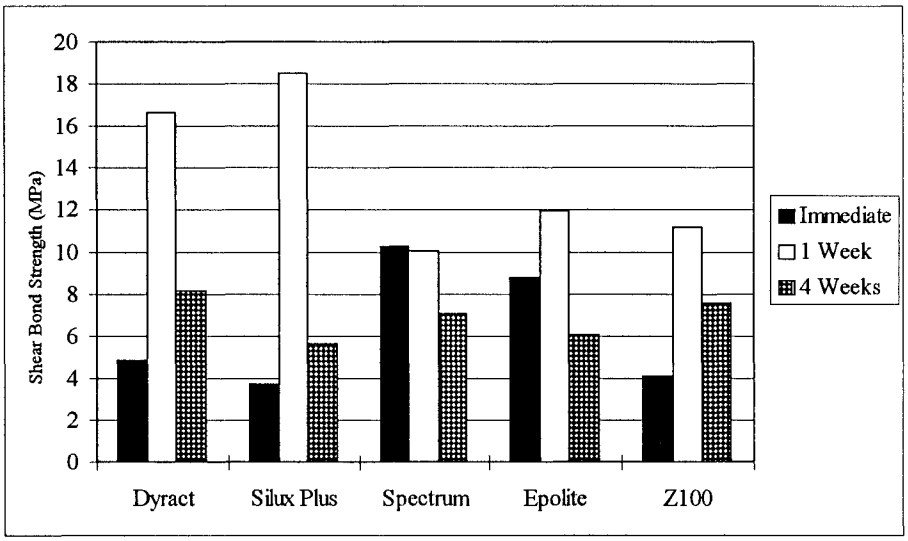

Figure 3. *Mean shear bond strength vs repaired samples at different time intervals*

Table 3. Comparison of Mean Shear Bond Strength of the Various Repaired Samples at Each Time Interval

Time after Repair	Differences
Immediate	Epolite > Silux Plus, Z100 Spectrum > Silux Plus, Z100, Dyract
1 week	No difference
4 weeks	No difference

Results of one-way ANOVA and Scheffé's Test ($P < 0.05$); > indicates statistical significance.

Table 4. Comparison of Mean Shear Bond Strength at the Various Time Intervals for Each Type of Repaired Samples

Materials	Differences
Dyract	1 week > immediate, 1 month
Silux Plus	1 week > immediate, 1 month
Epolite	1 week > 1 month
Spectrum	No difference
Z100 (control)	1 week > immediate

Results of one-way ANOVA and Scheffé's Test ($P < 0.05$); > indicates statistical significance.

At 1 week no significant difference was noted in the shear bond strength of all the repaired materials. The faster rate of cross-linkage formation in repaired Spectrum and Epolite may have used up the majority of the remaining unreacted methacrylate groups soon after repair. However, due to the slower rates of cross-linkage formation in the repair of Dyract, Silux Plus, and Z100, more unreacted methacrylate groups remained, and continual cross-linkage formation occurred through the first week. Thus, repaired Dyract, Silux Plus, and Z100 achieved similar shear bond strengths to repaired Spectrum and Epolite (Vankerckhoven & others, 1982).

The rate of further polymerization occurred at similar rates for all the repaired materials at the 4-week interval. This accounted for the insignificant difference in the repair shear bond strengths at that time.

The shear bond strength between Dyract and Z100 at 1 week was significantly greater than that after immediate repair and at the 4-week interval. This could be attributed to the components of polyalkenoate cement present in the material (Phillips, 1991). The complete curing cycle of the material occurs at about 1 week. Hence we would expect the strength to be greatest at 1 week. However, due to the water sorption that occurs during the period of storage, we would also expect the process of leaching to occur. This could result in the deterioration of the shear bond strength of the repair at 4 weeks.

For Silux Plus, results similar to Dyract were observed. An increase in the shear bond strength at the 1-week interval was noted due to the continued formation of cross-linkages between the methacrylate groups. The decrease in the shear bond strength at the 4-week period could be due to the water sorption occurring in the materials. The macromolecules are forced apart by the diffusion of the water, thus decreasing the strength of the material (Vankerckhoven & others, 1982; Ralph, 1991).

In the repair of Spectrum, the shear bond strength did not show significant changes up to the 4-week period. This showed that the repaired materials obtained their optimal strength almost immediately, and the repair had minimal leaching and water sorption up to 4 weeks after repair.

For Epolite, the optimal shear bond strength of its repair with Z100 was achieved almost immediately. This accounted for the insignificant difference between the repair shear bond strengths immediately after repair and at the 1-week interval. However, deterioration in the bond strength was noted at the 4-week interval, possibly due to the leaching out of unreacted methacrylate, resulting in the decrease in formation of cross-linkages. In addition, water sorption, which may also have occurred, could also weaken the matrix.

For the repair done on Z100, the 1-week shear bond strength was greater than that immediately after repair. This could be due to the continual formation of cross-linkages between the parent material and the adhesive. However, there is minimal polymerization and linkage deterioration from this point onwards, accounting for the insignificant difference between repair shear bond strengths at 1 week and 4 weeks after repair.

Studies have shown that the maximum shear bond stress anterior composite resin restorations would be subjected to was about 9 MPa (Farah & Craig,1974). From our results, the immediate mean shear bond strength of the repair of Spectrum and Epolite attained acceptable clinical strength. However, the repair of the other materials did not achieve acceptable immediate shear bond strength. The patient may best be advised not to stress the area of repair immediately after the procedure.

At 1 week, all the repairs matured to acceptable levels above the clinically required shear bond strengths. However, all the repaired materials

showed deterioration in the shear bond strength at the 4-week interval. These repair shear bond strengths were slightly below that of the clinically acceptable level postulated by Farah and Craig (1974).

Z100 achieved adequate shear bond strength when repaired on Spectrum, Dyract, and itself at the 4-week interval. The repair of Z100 on itself did not show significantly better shear bond strength than repairs done on the other study materials. The repair on Silux Plus and Epolite at 4 weeks was lower than the rest when compared to the clinically acceptable value. However, this was not statistically significant.

CONCLUSIONS

The shear bond strength between the various mature parent materials and the new hybrid composite resin (Z100) used for repair was minimally adequate for clinical service. Repair of Z100 to the parent material of the same type of composite resin did not produce significantly higher levels of shear bond strength. However, there was a general increase in the repair shear bond strengths at 1 week and general deterioration at the 4-week interval in most of the repaired materials.

(Received 27 January 1998)

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Microleakage of a Consolidated Silver Direct Filling Material

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Clinical Relevance
The microleakage associated with an experimental mercury-free consolidated silver is less than that measured with spherical and dispersed-phase amalgams. The consolidated silver also appears to perform well when using copal and polyamide cavity varnishes.

SUMMARY

Microleakage of an experimental direct filling material comprised of a chemically precipitated silver powder that had been surface treated with a dilute acid to promote cold welding upon consolidation was evaluated. Microleakage was compared to both dispersed-phase and spherical amalgam by use of an in vitro gas-diffusion method and in class 5 restorations placed in extracted human teeth. The effect of two cavity varnishes and two dentin adhesives as cavity liners on microleakage was also evaluated using extracted teeth. Microleakage of silver powder consolidated with dental instruments was less than that found with dental amalgam. The use of copal or polyamide cavity varnish resulted in the lowest combination of microleakage on dentin and enamel margins.

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INTRODUCTION

An experimental mercury-free direct filling alloy is made up of combinations of powdered metals, comprised mostly of silver, to produce a filling material very similar in handling properties to direct gold. The powdered metal is thermally annealed, chemically treated to remove surface oxides, then compressed to form a solid mass. Welding together of the individual oxide-free particles is accomplished by manual consolidation of the metal similar to cohesive gold at room or body temperature. The powders are delivered as a slurry or mixture of metal particles within a dilute acid that serves both to remove surface oxide and protect the particles from re-oxidizing

during consolidation. The powder-liquid mixture has a consistency and handling properties similar to freshly triturated amalgam. This material is intended to be incrementally consolidated directly into a cavity preparation using hand or mechanically assisted condensing instruments, much like those used for mercury amalgam (Dariel & others, 1995; Eichmiller & others, 1996).

One important consideration for all dental restorative materials is the ability to completely seal the tooth/filling interface. Amalgam exhibits a high degree of marginal leakage shortly after placement. Margin gaps ranging from 5 μm to 20 μm have been reported (Brännström, 1984), along with an average gap of 13 μm for dispersed-phase amalgam restorations (Mertz-Fairhurst & Newcomer, 1988). These gaps are thought to be due to shrinkage of the material as the mercury reacts with the alloy powder to chemically form intermetallic compounds. Cavity varnishes have historically been used on the cavity walls prior to placement of amalgam to provide a provisional seal of the interface. It has been reported (Ben-Amar, Cardash & Judes, 1995) that corrosion products from the amalgam eventually fill the margin gap, effectively sealing the interface. Unlike amalgam, the experimental direct filling alloy does not undergo any chemical reaction that results in dimensional changes once the alloy has been placed. This dimensional stability could result in better margin adaptation and less microleakage than conventional amalgam.

A dilute aqueous acid, fluoroboric acid (HFB_4), is used to deliver the powdered metal, and any reaction between the acid and the enamel or dentin has not been well characterized. Many types of acid are safely used on dentin and enamel as cavity etchants prior to placing composites and adhesives. One reason for the safe use of these acids is the excellent buffering ability of tooth mineral. The pH and concentration of the HFB_4 used with this alloy system is similar to currently used acids, but there is no provision for rinsing or removing the acid from the surface, as is common with most adhesive etching techniques.

The use of cavity liners and varnishes may reduce the contact of acid with the tooth and resulting demineralization. Less demineralization could lead to lower microleakage, or the varnish could provide a provisional seal of the margin. The low corrosion potential of this alloy system may also prevent formation of corrosion products at the restoration-tooth margin. These and other factors could have a profound effect on the resulting microleakage of the final filling.

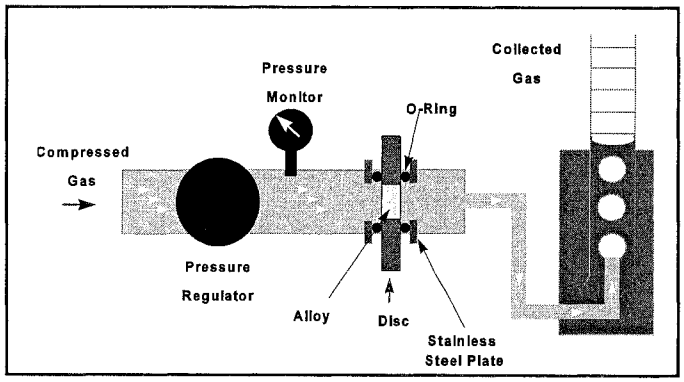

Figure 1. *Gas diffusion apparatus*

The purpose of this investigation was to evaluate the microleakage of a direct consolidated silver material. Microleakage was measured and compared to conventional amalgam in gas-diffusion experiments (Granath & Svensson, 1970) and in class 5 restorations placed in extracted teeth. Copal varnish, a polyamide liner, and a filled and unfilled bonding agent were evaluated in class 5 restorations for microleakage.

METHODS AND MATERIALS

Gas Diffusion Method

In the first set of experiments, microleakage was measured by using a gas-diffusion technique (Granath & Svensson, 1970) with an apparatus shown in Figure 1 that was similar to instrumentation used in published amalgam microleakage studies (Mahler & Nelson, 1984). This apparatus applied gas pressure (600 mm Hg) to one side of simulated class 1 cavity preparations placed in a Macor (Corning Inc, Corning, NY 14831) ceramic disk with no pulpal wall. The volume of gas leaking through the alloy-disk interface per unit time was measured and recorded as the microleakage index in mL/min. Three different alloy systems were compared: an admixed amalgam alloy (Dispersalloy, LD Caulk, Milford, DE 19963), a spherical amalgam alloy (Tytin, Kerr, Orange, CA 92867), and experimental consolidated silver alloy.

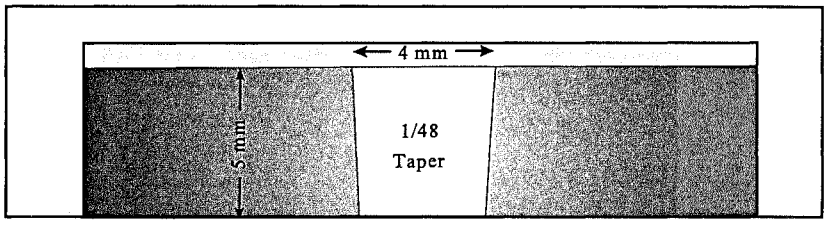

Figure 2. *Microleakage disk configuration*

Simulated cavity preparations consisted of holes 4 mm in diameter drilled in the center of disks 25 mm in diameter by 5 mm thick cut from Macor ceramic. The 4 mm hole was centered in the disk and was slightly tapered (1 part in 48) by use of a No 1 taper pin reamer (Figure 2). Thirty disks were prepared (10 for each material), and the fillings were placed in random order to minimize operator and environmental effects on microleakage. The ceramic disks were filled by placing the disk on an acrylic base during condensation to simulate a pulpal floor. This base had a slight recess to allow for overfilling of the disk at the pulpal floor. The amalgam alloys were triturated per manufacturer's instructions and then incrementally condensed into the cavity with a smooth-faced condenser 1.5 mm in diameter, stepping over the entire area of each increment in 10 even steps. Condensation was done on a load table with a manual load of 2.3 kg. The lower load was necessary to prevent excess extrusion of the amalgam around the plugger tip and out of the mold. Four increments were placed, slightly overfilling the cavity. Immediately after filling, the disks were removed from the base, and a new razor blade was used to remove excess amalgam, moving the blade from the edges inward to avoid burnishing of the margins.

Chemically precipitated silver powder (NIST laboratory batch CJ161) was thermally annealed in air for 2 hours at 450 °C and gently sifted through a 200-mesh sieve, resulting in an average particle size of approximately 150 μm. The powder was activated immediately prior to use as follows: 0.5 g of the silver powder was mixed with 350 mL of 10% HBF_4 (volume fraction as diluted from a 48% by mass concentrated HBF_4 solution) on a magnetic stirrer for 1 minute. The solution was allowed to settle for 2 minutes; the excess liquid was decanted, leaving the powder just covered with acid. The silver powder was delivered to the samples with a spatula and condensed into the disk with a 1.5 mm amalgam condenser with a serrated tip at a load of 3.5 kg. The disk was slightly overfilled and the excess immediately removed from both sides of the disk by wet polishing with 320-, 400-, and then 600-grit silicon carbide abrasive papers.

All samples were tested for microleakage at 0 hours, 24 hours, and 2 weeks of storage at 37 °C in an artificial saliva solution. Microleakage was measured by applying compressed nitrogen gas at 8.27 MPa (600 mm Hg) to one side of the sample disk and collecting the gas that leaked through the sample in an inverted graduated cylinder (Figure 1) over a known period of time. The leakage rate was recorded for each sample in mL/min. Representative samples from each of the three materials were then cross sectioned and polished, and the interface was studied by scanning electron microscopy.

In a second similar experiment, disks were fabricated from high-density polyethylene in place of the Macor ceramic. Polyethylene was chosen to eliminate any potential for a reaction to occur between the HBF_4 and the disk surface. Twenty-seven total samples were prepared, nine each of Dispersalloy, Tytin, and the consolidated silver. The filling, polishing, and testing were done in the same manner as for the Macor disks. Leakage rate measurements were made after 24 hours and 2 weeks of storage in artificial saliva at 37 °C. To determine any effect water may have in the leakage paths, nine additional samples of each material were fabricated and tested after 24 hours of immersion in 37 °C artificial saliva, tested again after 1 week of dry storage in air, and tested again after re-immersing for a second week in artificial saliva.

Class 5 Microleakage

Microleakage studies with extracted teeth were done in two parts. For both studies noncarious human teeth extracted for orthodontic reasons were stored in deionized water with a bactericidal agent, 0.2 % sodium azide (mass fraction), until ready for use. Residual tissue tags were scraped and the teeth thoroughly rinsed under running tap water for 15 minutes to remove sodium azide solution. Green stick modeling compound was used to seal the occlusal pit and fissures as well as the root apices.

In the first study, 60 teeth were prepared on the buccal surface for class 5 restorations using a high-speed handpiece with a #330 bur. Undercuts were placed in the occlusal and gingival walls with a #½ round bur for mechanical retention of the filling. The preparations were approximately 1 mm into dentin, oblong in shape, measuring 3 mm x 5 mm, paralleling the cementoenamel junction. The gingival half of the preparations was kept 1 mm occlusal to the cementoenamel junction. Cavity preparations were rinsed for 20 seconds with an air-water spray and gently air dried for 30 seconds. One coat of polyamide liner (Barrier, Teledyne/Getz, Ft Collins, CO 80553) was applied to preparations of samples that required a cavity liner. The silver powder was activated immediately prior to use as follows: 0.5 g of the silver powder was mixed with 350 mL of the 10% HBF_4 (volume fraction) on a magnetic stirrer for 1 minute. The solution was allowed to settle for 2 minutes, the excess liquid decanted, and 500 mL of 2% HBF_4 (volume fraction as diluted from a 48% by mass concentrated HBF_4 solution) added. This was mixed and allowed to settle for 1 minute and the excess again decanted, leaving the silver alloy just covered with liquid. The silver alloy was delivered to the samples with a spatula, and condensed with a serrated amalgam condenser 1.5 mm in diameter on

Table 1. Mean Microleakage Rates through Macor Disks

	Immediate	24 Hours	2 Weeks
Dispersed-phase amalgam	0.88 ± 0.85	0.77 ± 0.44	0.48 ± 0.31
Spherical amalgam	0.56 ± 0.38	1.74 ± 1.61	2.10 ± 2.25
Consolidated silver	0.01 ± 0.02	0	0

Mean microleakage rate in mL/min (mean ± standard deviation, n = 10)

a load table at 3.5 kg. The amalgam (Dispersalloy) was triturated and condensed following the manufacturer's recommendations. Each restoration was overfilled and burnished, and the amalgam was carved back to contour with a Hollenback carver. After setting for 24 hours, the restorations were polished flush with the tooth surface by use of disks (Sof-Lex, 3M Dental Products, St Paul, MN 55144) and a slow-speed handpiece.

Four groups of fillings were investigated with 15 preparations included in each group. The groups consisted of consolidated silver placed with no liner in the cavity or over a polyamide liner, and dispersed-phase amalgam with and without a polyamide liner. The design layout was randomized with regard to restoration material and liner. Restorations were placed in random blocks of four to ensure that all environmental variables were equally distributed across the four groups.

All specimens were stored at room temperature in an artificial saliva-like solution except during processing. All teeth were thermally stressed for 3000 cycles between 5 °C and 55 °C with a dwell time of 1 minute at each temperature. After thermocycling, the teeth were placed in a 0.5% solution of methylene blue (mass fraction) for 24 hours. The specimens were rinsed and then cut longitudinally through the center of the restorations using a 50 μm diamond sectioning saw. Each specimen half was incrementally polished on a rotary polishing wheel with 1200- grit, 2400-grit, and 4000-grit wet silicon carbide paper. The sections were then viewed under light microscopy at X40 magnification, scored for microleakage, and photographed.

Microleakage scores were based on the degree of dye penetration according to the following scale: 0 = no leakage; 1 = dye penetration in enamel only, or in first 0.5 mm of gingival dentin; 2 = dye penetration past the dentinoenamel junction, or beyond 0.5 mm of gingival ; 3 = dye penetration to the axiogingival line angle; 4 = dye penetration up the axial wall of the preparation. Comparison was done by chi-square analysis of the scores for each material.

The second microleakage experiment required 75 cavity preparations distributed into five groups with 15 preparations in each group. The preparations were done in the same manner and dimensions as previously described, with the exception that the gingival margin was located 1 mm apical to the cementoenamel junction. All of the fillings were consolidated silver placed over four different lining materials or control (no liner). The design layout was randomized with regard to the liner used to ensure that all environmental variables were equally distributed across the groups. Group 1 did not have any type of liner. Group 2 had two coats of copal cavity varnish (Copalite, Bosworth, Skokie, IL 60076). Group 3 had two coats of polyamide liner (Barrier). Group 4 had a polymer adhesive that did not contain any type of particulate filler (All-Bond 2, Bisco Dental Products, Itasca, IL 60143) placed according to the manufacturer's instructions. Group 5 had an adhesive containing polymethylmethacrylate particulate filler (Amalgambond Plus, Parkell, Farmingdale, NY 11735) placed according to the manufacturer's instructions. Storage, thermocycling, staining, and section preparation were done in the same manner as the previous experiments. Dye penetration was scored separately for the occlusal enamel margins and the gingival dentin margins using the same 0-to-4 scale.

RESULTS

Gas Diffusion Method

Microleakage results showing gas diffusion rates through restorations placed in the ceramic disks are shown in Table 1. When tested immediately after placement, the difference in average leakage between amalgam and the consolidated silver was statistically significant (t-test, $P \leq 0.01$), but there was no significant difference between the dispersed-phase and spherical amalgams ($P = 0.68$). After 24 hours of storage in artificial saliva, the difference in average leakage between both types of amalgam and the consolidated silver was still significant ($P \leq 0.01$), but there was no significant difference between the two amalgams ($P \geq 0.1$). After 2 weeks of storage in artificial saliva, the difference in leakage between both amalgams and the consolidated silver was statistically significant ($P \leq 0.01$), but there was now a significant difference between the two types of amalgam ($P = 0.04$). A regression analysis of leakage for the dispersed-phase amalgam over the 2-week period indicated that there was no statistical significance ($P = 0.13$) to the slight downward trend in leakage rate with time. A similar analysis of the

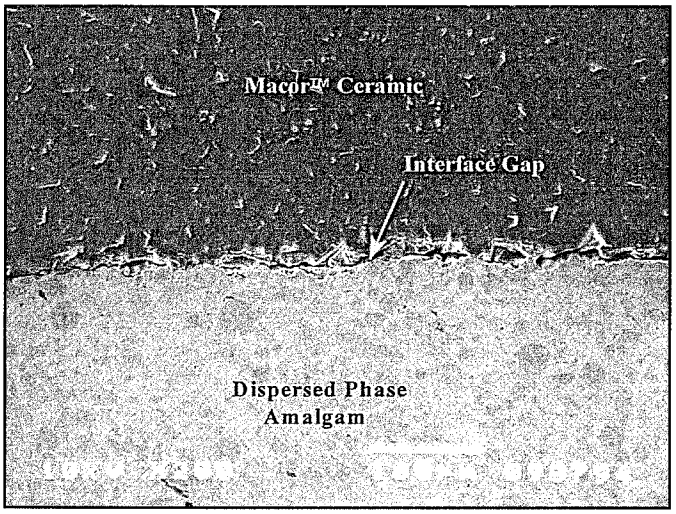

Figure 3. *Scanning electron micrograph of dispersed-phase amalgam in a Macor ceramic disk (magnification X29.6)*

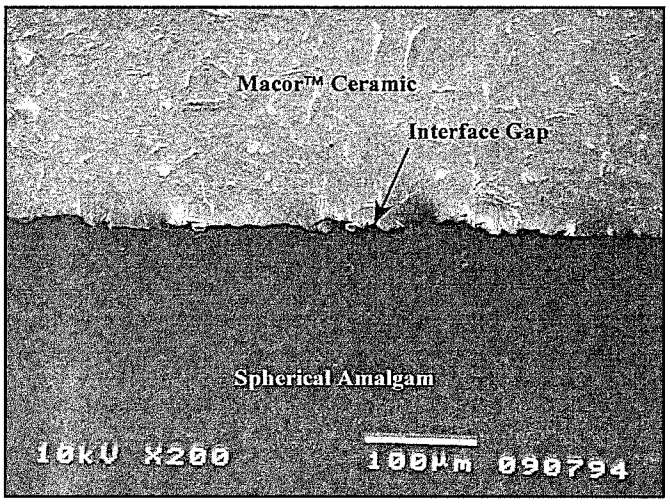

Figure 4. *Scanning electron micrograph of spherical amalgam in a Macor ceramic disk (X29.6)*

spherical amalgam indicated a slight trend of increasing leakage with time, but was not significant ($P = 0.06$). The consolidated silver showed no measurable leakage at the 24-hour and 2-week time periods, and no time-related trend could be determined ($P = 0.35$).

Scanning electron microscopy of the cross sections of the dispersed-phase and spherical amalgam restorations showed interfacial gaps between the alloy and the Macor that were presumably due to setting shrinkage of the alloy. The gaps were continuous along the entire length of the interface in both alloy types (Figures 3 & 4). Similar cross sections of the consolidated silver showed close adaptation of the metal to the ceramic with very little indication of a continuous interfacial gap (Figure 5). Some porosity was evident in the consolidated silver both in the body of the metal and at the interface, but the porosity appeared to have little intercommunication. An altered zone of approximately 75 µm in width was evident in the ceramic, starting at the interface and proceeding into the body of the ceramic. This alteration was not evident in the amalgam samples and was presumably due to a reaction between the 10% fluoroboric (volume fraction) acid and the ceramic. These experiments were repeated in the second part using high-density polyethylene disks to eliminate any effect this reaction may have had on leakage and to attempt to simulate the resilience of dentin with the mold material.

The results of the high-density polyethylene disk experiments were considerably different from those in the ceramic. Overall leakage was much greater for

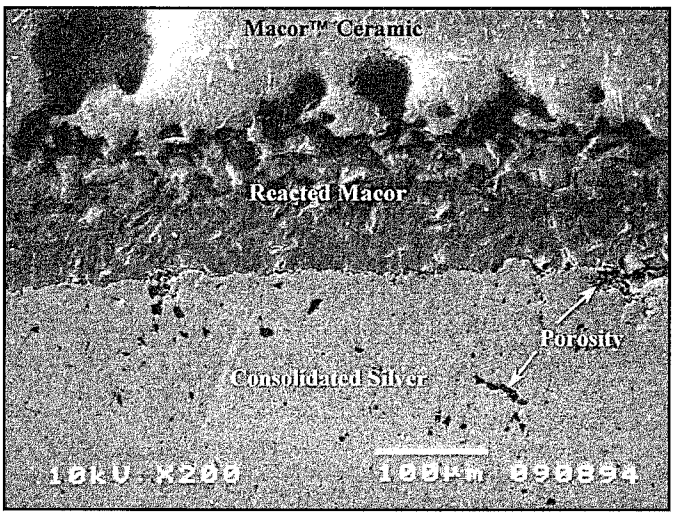

Figure 5. *Scanning electron micrograph of consolidated silver in a Macor ceramic disk (X29.6)*

Table 2. Mean Microleakage Rate in High-Density Polyethylene Disks

	24 Hours	2 Weeks
Dispersed-phase amalgam	27.7 ± 4.7 (n = 9)	17.1 ± 3.5 (n =9)
Spherical amalgam	22.4 ± 5.6 (n = 7)	6.9 ± 4.1 (n = 8)
Consolidated silver	2.6 ± 4.7 (n = 9)	4.3 ± 3.5 (n = 9)

Mean microleakage rate in mL/min (mean ± standard deviation)

Table 3. *Mean Microleakage Rate in Dried and Reimmersed High-Density Polyethylene Disks*

Storage Conditions	Dispersed-Phase Amalgam	Spherical Amalgam	Consolidated Silver
24-hour dry (mL/min)	5.8 ± 0.6 (n = 8)	1.5 ± 0.6 (n = 9)	0.1 ± 0.6 (n = 9)
1-week dry (mL/min)	7.6 ± 1.6 (n = 9)	6.0 ± 1.6 (n = 9)	7.8 ± 1.6 (n = 9)
2-week reimmersion (mL/min)	22.5 ± 6.4 (n = 9)	27.5 ± 7.5 (n = 9)	0.9 ± 0.3 (n = 9)

all three materials, as shown in Table 2. Several conventional amalgam samples were eliminated from the statistical analysis by Cook's Outlier *t*-testing, due to the extreme amount of microleakage measured in those samples. At the 24-hour time interval, both the dispersed-phase and the spherical alloys leaked significantly more than the experimental alloy (ANOVA and *t*-tests, $P < 0.05$), but the dispersed-phase alloy was not significantly different from the spherical alloy. At the 2-week time interval, the dispersed-phase alloy leaked significantly more than the spherical and experimental alloys (ANOVA and *t*-tests, $P < 0.05$). Some trends were noted over the 2-week period: the admixed and spherical alloys leaked less as time progressed, and leakage variability between samples increased. The experimental alloy in the polyethylene, unlike the experimental alloy in ceramic, demonstrated some leakage at both the 24-hour and 2-week intervals in several samples.

To test the hypothesis that the low microleakage results associated with the consolidated silver may have been due to liquid retained in microporosities at the alloy/polyethylene interface, a second set of polyethylene samples was stored in a dry environment at 37 °C for 1 week to allow any accessible liquid to evaporate. The dried samples were then placed back in artificial saliva for an additional 2 weeks and retested for microleakage. Results from this study are summarized in Table 3. The microleakage rate for the consolidated silver increased from 0.1 ± 0.6 mL/min at 24 hours to 7.8 ± 1. 6 mL/min after drying for 1 week. After reimmersion in artificial saliva for 2 weeks, however, the consolidated silver leakage rate diminished to 0.9 ± 0.3 mL/min. Dry storage of both amalgam alloys resulted in a minimal increase in the leakage rate, but reimmersion for 2 weeks dramatically increased the leakage rates. The reimmersion rates for silver and dispersed-phase amalgam were closer to those achieved in the earlier set of samples in which no drying was done. Light microscopy of cross sections of several of the filled disks revealed that all of the alloys showed exceptional adaptation to the preparation wall (Figures 6-8). There was no reactive layer evident at the interface in the silver samples consolidated in the polyethylene disks.

Class 5 Microleakage

The first class 5 microleakage experiment compared consolidated silver to dispersed-phase amalgam in cavities with and without a polyamide varnish liner. The number of specimens and their associated dye microleakage rank scores for each filling material and liner are given in Table 4. The

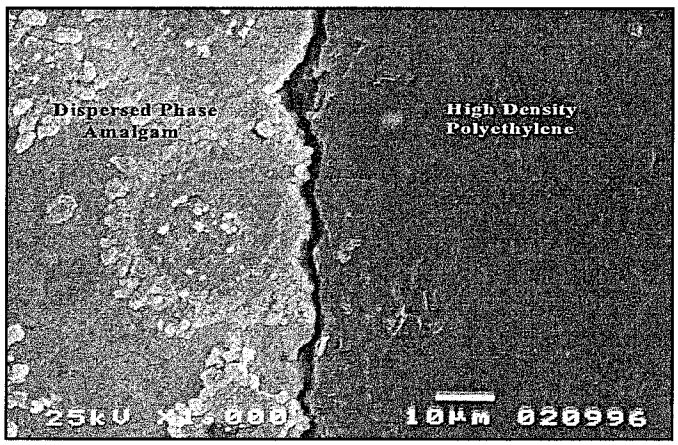

Figure 6. *Scanning electron micrograph of dispersed-phase amalgam in a high-density polyethylene disk (X29.6)*

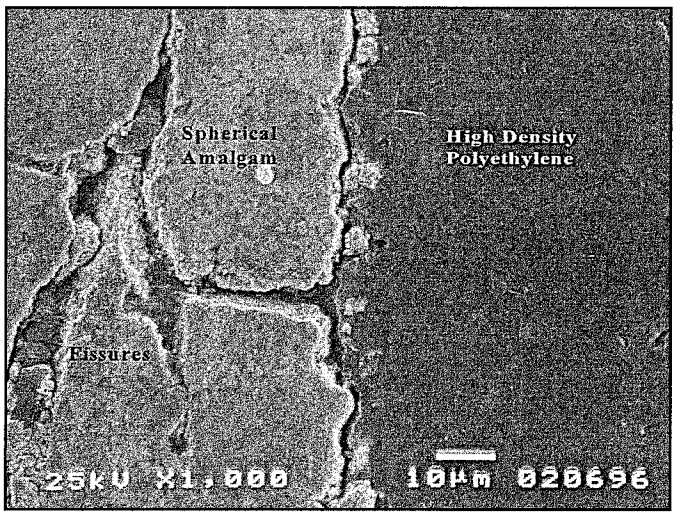

Figure 7. *Scanning electron micrograph of spherical amalgam in a high-density polyethylene disk (X29.6)*

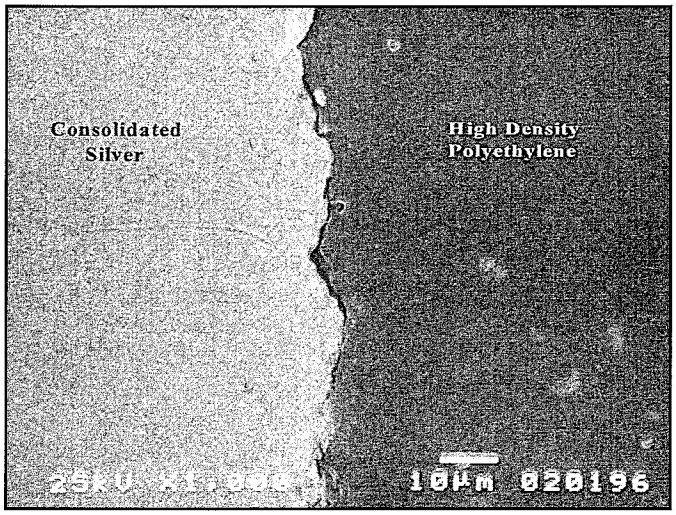

Figure 8. *Scanning electron micrograph of consolidated silver in a high-density polyethylene disk (X29.6)*

median values of leakage were 0 for the consolidated silver without the liner, 0 for the consolidated silver with the liner, 4 for the amalgam without the liner, and 0 for the amalgam with the liner. The fraction of specimens with a severe grade 4 leakage in each group was 6.7 % for the consolidated silver without a liner, 13.3 % for the consolidated silver with a liner, 93.3 % for amalgam without the liner, and 46.7% for amalgam with a liner. Chi-square analysis ($P = 0.001$) indicated no difference in leakage between the consolidated silver with or without the liner, and the silver leakage was significantly different from the amalgam either with or without the liner. There was also a significant difference in leakage between amalgam with a liner and amalgam without a liner.

Results of the second tooth microleakage trial studying the effects of different cavity liners on leakage in enamel margins are shown in Table 5 and for dentin margins in Table 6. The median values of leakage rank for enamel margins were: with no liner = 0; for enamel margins lined with copal varnish = 0; for enamel lined with polyamide liner = 0; for enamel lined with an unfilled dentin adhesive = 2; and for enamel lined with a filled adhesive = 0. Chi-square analysis indicated that enamel leakage was liner dependent ($P = 0.017$) with the unfilled dentin adhesive demonstrating greater leakage than any of the other materials. The median values for dentin leakage were: no liner = 2; copal varnish = 1; polyamide liner = 1; unfilled dentin adhesive = 2; and filled dentin adhesive = 2. Chi-square analysis indicated that the leakage was again liner dependent ($P < 0.001$), with both the copal varnish and polyamide liner resulting in the least leakage. Leakage was statistically greater in dentin than enamel (Chi-square, $P < 0.01$) when no liner was used and when using copal varnish. For the other materials leakage was not dependent upon the margin location.

DISCUSSION

The microleakage rates measured in ceramic for the amalgam were similar to those reported by Mahler and Nelson in 1994. Slight leakage was measured in only three of the 10 consolidated silver samples measured immediately after placement. This leakage disappeared after 24 hours and was not present at the 2-week measurement. Microscopy of the silver/ceramic interface indicated very close lateral adaptation of the alloy to the ceramic surface, very few interfacial gaps, and no gaps that traversed the entire length of the margin. The close adaptation was in strong contrast to the amalgam

Table 4. Microleakage Scores for Class 5 Restorations

	Consolidated Silver No Liner	Consolidated Silver Polyamide Liner	Amalgam No Liner	Amalgam Polyamide Liner
Leakage score = 0	13	12	0	8
Leakage score = 1	0	0	0	0
Leakage score = 2	1	0	0	0
Leakage score = 3	0	1	1	0
Leakage score = 4	1	2	14	7

Number of restorations with each leakage ranking score

Table 5. Microleakage Scores for Class 5 Consolidated Silver Restorations in Enamel Margins

	No Liner	Copal Varnish	Polyamide Liner	Adhesive No Filler	Adhesive with Filler
Leakage score = 0	12	11	9	4	8
Leakage score = 1	3	3	4	2	4
Leakage score = 2	0	1	2	7	3
Leakage score = 3	0	0	0	2	0
Leakage score = 4	0	0	0	0	0

Number of restorations with each leakage ranking score

samples, where gaps were consistently present and traversed the entire length of the margin. The consolidated silver is a cold-welded material that does not undergo the mercury-intermetallic matrix-forming reactions responsible for the setting of amalgam and the associated setting shrinkage. This may have been the reason for this material maintaining close adaptation to the ceramic and low leakage values.

Scanning electron microscopy of the silver/ceramic interface revealed a wide zone of altered ceramic adjacent to the interface. The zone was approximately 75 μm in width and had a different contrast, but a crystalline microstructure appearing similar to the bulk of the ceramic. This zone may have been the result of the HBF_4 reacting with the glassy matrix of the ceramic. Samples later consolidated using a slurry of silver in a solution with a lower concentration of HBF_4 (2 % volume fraction HBF_4) showed a similar but narrower reaction zone. There was concern that dissolution and reprecipitation of the ceramic may have been responsible for the closely adapted interface. The experiments were repeated using high-density polyethylene disks to eliminate this possibility. The leakage rates measured in high-density polyethylene were more than 10 times higher than those measured in ceramic. The relative leakage of the consolidated silver was once again much lower than either of the amalgams. It was noted during condensation of amalgam that the freshly triturated material had a tendency to slide up the walls of the preparation and fold over onto the top of the previously condensed material. This

Table 6. Microleakage Scores for Class 5 Consolidated Silver Restorations in Dentin Margins

	No Liner	Copal Varnish	Polyamide Liner	Adhesive No Filler	Adhesive with Filler
Leakage score = 0	1	2	4	0	4
Leakage score = 1	6	9	8	3	2
Leakage score = 2	1	2	2	9	4
Leakage score = 3	5	2	1	0	5
Leakage score = 4	2	0	0	3	0

Number of restorations with each leakage ranking score

was encountered very frequently with the more plastic spherical amalgam than with the dispersed-phase amalgam. Microscopic observations of polished cross sections of spherical amalgam samples revealed frequent fissuring and layering of the material with orientation of the layers being parallel to the wall of the polyethylene mold. These fissures may have contributed to the leakage, but it could not be confirmed that any individual fissure line communicated completely through the sample. A shrinkage gap was observed at the metal/polyethylene interface similar in width to that found in the ceramic samples. Once again, the consolidated silver appeared to be closely adapted to the polyethylene cavity wall. There was no obvious explanation for the higher overall leakage rates observed in the polyethylene, but the elastic modulus of the material was much lower than the ceramic and may have resulted in a less stable substrate against which to condense the materials.

Another possible reason for the low leakage values with the consolidated silver could be that residual acid could be retained in defects at the alloy/disk interface, preventing the gas from diffusing through the interface. To test this hypothesis disks were filled, measured after 24 hours of immersed storage, dried for 1 week to remove residual moisture, and re-immersed for 2 weeks to attempt to re-imbibe the interface with fluid. In this experiment, the dry microleakage rate for both amalgams was much lower than that measured after reimmersion for 2 weeks. The reason for this change is not clear but may be related to micromovement of the sample within the tapered polyethylene during storage or testing. Dry storage for 1 week increased the microleakage of the consolidated silver, but leakage was dramatically reduced by reimmersion. This reduction could have been due to the formation of corrosion products in the interface, but was more likely due to fluid refilling the microporosity defects along the interface, inhibiting the flow of the gas.

The class 5 microleakage evaluation of amalgam and consolidated silver showed that the ranking of leakage observed in the gas diffusion studies correlated well with the dye leakage in human teeth. Again, the silver restorations ranked lower in leakage than the amalgam restorations. Adaptation to all walls of the cavity preparation appeared comparable, even though the direction of force for condensation was primarily against the axial wall of the preparation. No obvious layering could be seen resulting from incremental filling for any of the materials. The use of a polyamide liner did not affect the leakage of the silver restorations, but did reduce the leakage of the dispersed-phase amalgam restorations. The liner layer was evident at the interface of both the amalgam and silver samples, but there were localized regions along the interface where the liner was removed, presumably from scraping by the condensing instrument. The preparations in this trial were completely surrounded by enamel margins and did not indicate how well the materials would seal against a dentin or cementum margin.

The second class 5 microleakage experiment used only consolidated silver with several different cavity lining materials. Both dentin and enamel margins were included in these preparations. The leakage results of silver against enamel without a liner and with the polyamide liner were very similar to the same combinations tested in the first experiment. In enamel margins, unfilled adhesive-lined restorations appeared to have considerably greater leakage. Leakage was also fairly high in the corresponding dentin margins. Several cross sections showed loss of the adhesive film from the surface of the cavity wall, and in some sections small inclusions of resin were observed within the mass of the consolidated silver. The resin observed within the hybrid layer was still intact.

The unfilled adhesive system was made up largely of the hydrophilic bifunctional monomer biphenyl-dimethacrylate. The acid used in this alloy system may react with this highly hydrophilic material, causing breakdown or dissolution, as was reported previously with acid etching of dentin adhesives (Erickson, 1992). The filled adhesive system had less leakage than the unfilled adhesive system (Tables 5 & 6). This may have been due to better resistance of the polymer to the HBF_4 acid or the formation of a thicker, more completely polymerized, layer on the cavity wall.

Microleakage in dentin was generally higher than that found in enamel, consistent with most reported microleakage studies. When considering placing consolidated silver in both enamel and dentin, both varnishes should provide a combination of low microleakage in all margins. The data show that filling a cavity without any liner can result in a good enamel seal, but may create a greater potential for leakage in dentin (Tables 5 & 6). The greater leakage seen with the adhesive resins indicates that using adhesive resin liners in combination with this consolidated silver should be investigated more thoroughly.

The direct consolidation and cold-welding process within the silver material may provide some explanation for the observed results. Loading during consolidation would be expected to cause elastic compression of the substrate due to its elastic modulus. With the silver, the cold-welding during this consolidation would not allow the filling to relax and flow back as the substrate rebounds after compression. The result could be an interface with residual compressive stresses maintaining the seal.

The more plastic amalgam would flow and deform as the substrate rebounds, leaving a neutral interface that opens when the amalgam shrinks. Enamel has a higher elastic modulus than dentin, resulting in higher interfacial stresses upon consolidation and a better seal than the lower modulus dentin.

The consolidated silver is highly dependent upon consolidation energy to achieve adequate cold-welding of the powder for strength. Cold welding of silver powder has been demonstrated at static stress levels as low as 7.1 MPa, and more complete densification and strength were achieved by the addition of impact energy (Eichmiller & others, 1995). This translates into a requirement for thorough consolidation using small plugger tips and thinner increments than those usually used with amalgam. A consolidated silver filling requires approximately twice as many increments and twice the consolidation time of conventional amalgam. Some advantages are that the silver can be polished and placed under load immediately after consolidation. Also, postoperative sensitivity normally associated with microleakage should be much lower with this material.

CONCLUSIONS

Microleakage measured by gas diffusion through ceramic and high-density polyethylene is low for consolidated silver when compared to dispersed-phase or spherical amalgam.

Enamel microleakage in class 5 cavities on extracted human teeth was found to be very low for consolidated silver, and remained low when cavity walls were lined with either copal varnish, polyamide liner, or a filled dentin adhesive. Enamel leakage increased when cavity walls were lined with an unfilled dentin adhesive.

Dentin microleakage was generally higher than enamel microleakage with consolidated silver, but was still relatively low when the cavity walls were lined with a copal varnish or a polyamide liner. Using either copal varnish or polyamide liner should provide low microleakage in both dentin and enamel when using consolidated silver.

Acknowledgments

This investigation was supported in part by USPHS Research Grant P50 DE09322 to the American Dental Association Health Foundation from the National Institutes of Health-National Institute of Dental Research and is part of the dental research program conducted by the National Institute of Standards and Technology in cooperation with the American Dental Association Health Foundation.

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.

(Received 12 February 1998)

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Self-Etching Primer vs Phosphoric Acid: An Alternative Concept for Composite-to-Enamel Bonding

M HANNIG • K-J REINHARDT • B BOTT

Clinical Relevance
Measurement of shear bond strength and analysis of marginal adaptation in class 2 composite resin restorations indicate that self-etching priming agents are effective in composite-to-enamel bonding without phosphoric acid pretreatment.

SUMMARY

The purpose of this in vitro study was (1) to investigate the composite-to-enamel bond strength and (2) to analyze the marginal adaptation of resin composite restorations in class 2 cavities using three self-etching priming agents in comparison to conventional phosphoric acid etching and bonding application. In the first part of the study 24 extracted bovine incisors were embedded in acrylic resin and ground flat with 800-grit paper. The following three self-etching priming agents/ composite resins were applied to the enamel surfaces of six teeth each: Clearfil Liner Bond 2/ Clearfil AP-X (Group I), Etch & Prime 3.0/Degufill mineral (Group II), Resulcin AquaPrime + MonoBond/Ecusit (Group III). In Group IV Ecusit-Mono/Ecusit was used after enamel etching with phosphoric acid (37%). Shear bond strength values measured on a T22 K testing machine at a crosshead speed of 1 mm/min were: 24.2 ± 3.0 MPa (Group I), 21.9 ± 1.4 MPa (II), 34.0 ± 3.6 MPa (III), and 26.3 ± 1.8 MPa (IV). ANOVA revealed significant ($P < 0.05$) differences in shear bond strength between groups, except comparison of Group I and II, and Group I and IV. In the second part of the study 24 standardized class 2 cavity preparations with the approximal box extending 1 mm above the CEJ were prepared in extracted human molars. Enamel margins were beveled and the teeth were divided into four groups of six teeth each. Cavities were restored using the self-etching priming agents Clearfil Liner Bond 2 (Group I), Etch & Prime 3.0 (Group II), and Resulcin AquaPrime + MonoBond (Group III). In Group IV composite resin restorations were placed after 37% phosphoric acid etching and bonding application (Ecusit-Mono). Quantitative SEM analysis of the marginal adaptation of the restorations after thermocycling (5-55 °C, 2500 cycles) and mechanical loading (100 N, 500,000 cycles) revealed excellent, gap-free margins in 91.2% (Group I), 93.0% (Group II), 92.0% (Group III), and 92.5%

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(Group IV) of the restorations' approximal area. There were no statistically significant differences between the four groups ($P < 0.05$). In conclusion, results of the present in vitro study indicate that use of self-etching primers may be an alternative to conventional phosphoric acid pretreatment in composite-to-enamel bonding restorative techniques.

INTRODUCTION

In 1955, Buonocore introduced the so-called acid-etch technique, which enabled the bonding of resin composites to the enamel surface. Buonocore found that acrylic resin could be bonded to human enamel that was conditioned with 85% phosphoric acid for 30 seconds. Today, most commercially available etchants contain 30% to 40% phosphoric acid, which provides enamel surfaces with the most retentive appearance (Silverstone & others, 1975). Acid etching creates a porous enamel surface layer ranging in depth from 5 to 50 μm. A low-viscosity bonding agent is used to penetrate the microporosities created in the enamel surface. After polymerization of the resin bonding agent, a durable attachment to the enamel is achieved by micromechanical retention. Bonding materials are generally unfilled resins based on bisphenol A glycidyl dimethacrylate (BIS-GMA) with the addition of diluents (such as triethyleneglycol dimethacrylate). In spite of the presence of two hydroxyl groups, the BIS-GMA monomer is insufficiently hydrophilic to compete with water for interaction with the enamel surface (Peutzfeldt, 1997). Therefore, the enamel surface must be carefully air dried after phosphoric acid etching in order to ensure that the bonding material completely wets the etched surface. Placement of composite restorations using the acid-etch technique has been investigated in numerous in vitro and in vivo studies and is considered a clinically established restorative method, provided that all steps of enamel conditioning (acid etching, water rinsing, air drying, application of the bonding agent) are properly executed. The enamel pretreatment could be simplified by the use of hydrophilic, acidic monomers capable of etching and penetrating the enamel simultaneously. Monomers with these properties are employed as self-etching primers in various dentin adhesives. Recently, some manufacturers have also offered them for enamel conditioning without phosphoric acid etching. The reactive components in self-etching primers are esters from bivalent alcohols with methacrylic acid and phosphoric acid or derivates (Table 1; Figure 1). The phosphate residue is thought to etch the enamel, while the methacrylate component of the molecule is

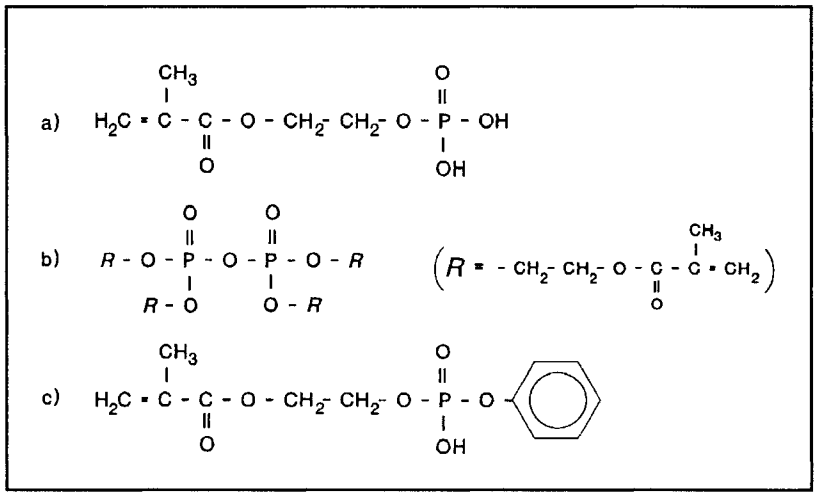

Figure 1. *Structural formula of reactive molecules used in self-etching priming agents: (a) 2-methacryloyloxyethyl-dihydrogenphosphate, (b) tetra-methacryloyloxyethyl-pyrophosphate, (c) 2-methacryloyloxyethyl-phenyl-hydrogenphosphate*

available for copolymerization with the bonding agent and composite resin. With this process, there is no need to rinse off reaction products or residual phosphoric acid ester, because both are subsequently polymerized into the bonding layer. However, up to now, only little data have been published that offer evidence that a self-etching primer could be used for composite-to-enamel bonding without previous phosphoric acid treatment (Gordan, Vargas & Cobb, 1997; Perdigao & others, 1997; Schmit & Van Sint Jan, 1997). The purpose of the present in vitro investigation was (1) to measure the composite resin-to-enamel shear bond strength after enamel conditioning with three self-etching priming agents, (2) to analyze the etching effect of the self-etching priming agents at the enamel surface, and (3) to evaluate the marginal adaptation of composite resin restorations in class 2 cavities conditioned with self-etching primers in comparison to enamel etching with phosphoric acid.

METHODS AND MATERIALS

Measurement of Shear Bond Strength

Shear bond strength measurement of the four bonding agents and composite resins listed in Tables 1 and 2 took place on bovine enamel. The labial surfaces of 24 crowns that had been cut off from freshly extracted bovine incisors stored in water were affixed to a glass plate and embedded in self-polymerizing acrylic resin (Technovit; Kulzer, Wehrheim, Germany). The pulp chamber was blocked

Table 1. Enamel Bonding Agents Used in Experimental Groups I-IV

GROUP	PRODUCT (MANUFACTURER) COMPONENTS	PRINCIPLE INGREDIENTS (According to Manufacturers)	MODE/STEPS OF APPLICATION
I	**Clearfil Liner Bond 2 (Kuraray Co, Osaka, Japan)**		
	LB PRIMER liquid A Lot #41134	2-methacryloyloxyethyl-phenyl-hydrogen-phosphate, n-methacryloyl-5-aminosalicylic acid	Mix LB PRIMER liquid A & liquid B. Apply to enamel for 30 seconds. Air blow gently.
	LB PRIMER liquid B Lot #41134	hydrophilic dimethacrylate, 2-hydroxyethylmethacrylate, ethanol, water	Apply LB Bond. Air blow gently. Light cure for 20 seconds.
	LB BOND Lot #41178	10-methacryloyloxydecyl-dihydrogen phosphate, 2-hydroxyethylmethacrylate, hydrophobic dimethacrylate, bis-phenol A diglycidyldimethacrylate, SiO_2	
II	**Etch & Prime 3.0 (Degussa AG, Hanau, Germany)**		
	Etch & Prime 3.0 Universal Lot #059703	2-hydroxyethylmethacrylate, ethanol, water	Mix Etch & Prime 3.0 Universal and Catalyst. Apply to enamel for 30 seconds. Air blow gently. Light cure for 10 seconds.
	Etch & Prime 3.0 Catalyst Lot #059703	tetra-methacryloyloxyethylpyro-phosphate, 2-hydroxyethylmethacrylate	Repeat the above mentioned steps.
III	**Resulcin AquaPrime + MonoBond (Merz Dental, Lütjenburg, Germany**		
	AquaPrime Lot #97200061	2-methacryloyloxyethyl-dihydrogen-phosphate	Mix AquaPrime with water (1:1). Scrub into the enamel surface for 30 seconds.
	MonoBond Lot #97200061	bis-phenol A diglycidyldimethacrylate, triethylenglycoldimethacrylate, polymethacryl-oligomaleic acid	Gently air dry. Apply MonoBond. Air blow gently. Light cure for 20 seconds.
IV	**Ecusit-Mono (DMG, Hamburg, Germany)**		
	Ecusit-Etch Lot #96510183	37% phosphoric acid	Etch enamel for 45 seconds. Rinse with water spray for 45 seconds. Thoroughly air dry.
	Ecusit-Mono Lot #96400066	bis-phenol A diglycidyldimethacrylate, triethylenglycoldimethacrylate, poly-methacrylated oligo-maleic acid	Apply Ecusit-Mono to etched enamel. Air blow gently. Light cure for 20 seconds.

Table 2. Composite Resins Used in Groups I - IV

Group	Bonding Agent	Composite Resin (Manufacturer)
I	Clearfil Liner Bond 2	Clearfil AP-X (Kuraray Co, Osaka, Japan) Lot #0309 A
II	Etch & Prime 3.0	Degufill Mineral (Degussa AG, Hanau, Germany) Lot #301
III	Resulcin AquaPrime + MonoBond	Ecusit-Composite (DMG, Hamburg, Germany) Lot #95470025
IV	Ecusit-Mono	Ecusit-Composite (DMG) Lot #96500100

with cement to prohibit any infiltration of the monomer into the dentin. Labial aspects of the crowns were ground on a wet-grinding disk (800-grit) to expose an enamel surface large enough for the shear bond test. Specimens were divided into four groups (I to IV) with six specimens each. In Groups I through III the enamel was conditioned by application of the self-etching bonding agents Clearfil Liner Bond 2 (Group I), Etch & Prime 3.0 (Group II), and Resulcin AquaPrime MonoBond (Group III) (Table 1). Enamel etching with phosphoric acid did not take place in these groups. The application of the bonding agents and the subsequent light curing adhered strictly to the manufacturers' instructions (Table 1). For control purposes, in Group IV enamel surfaces were treated according to the conventional acid-etch technique: the enamel was etched with a 37% phosphoric acid gel (Ecusit-Etch) for 45 seconds. After thoroughly rinsing off the phosphoric acid gel, a thin layer of bonding material (Ecusit-Mono) was applied and light cured in Group IV (Table 1). Glass tubes with a diameter of 4 mm were mounted on the pre-treated enamel surfaces to apply the composite resins (Table 2). The glass tubes could be easily removed after 40 seconds of light curing (Translux EC; Kulzer, Wehrheim, Germany) of the composite materials. The test objects obtained by this procedure were stored in water at 37 °C for 24 hours prior to the shear bond test. After the specimens had cooled to room temperature, their shear bond strength was measured on a T 22K testing machine (J J Lloyds Instruments, Gerlingen, Germany) at a crosshead speed of 1 mm/min. The debonded enamel surfaces were evaluated in a stereomicroscope at X40 magnification to assess the composite-enamel fracture pattern. Statistical analysis of comparisons among the materials was achieved by analysis of variance and Student-Newman-Keuls test. The level of significance was set at $P < 0.05$.

SEM Analysis of the Enamel Etching Pattern

Of the four materials listed in Table 1, three are described by the manufacturer as self-etching bonding agents: Clearfil Liner Bond 2, Etch & Prime 3.0, and Resulcin AquaPrime MonoBond. In order to test the self-etching effect, the enamel surfaces of extracted human molars were ground on wet-grinding disks (Jean Wirtz GmbH, Düsseldorf, Germany), using grit sizes down to 4000. The priming components of the three self-etching bonding agents (LB PRIMER liquid A and B, Etch & Prime 3.0 Catalyst and Universal, Resulcin AquaPrime) were each applied to six of these enamel surfaces according to the reaction times specified by the manufacturer (Table 1) but were not light cured. Subsequently, the areas were thoroughly rinsed with alcohol and acetone. After drying, the specimens were gold sputtered and photographed in a scanning electron microscope.

SEM Analysis of the Marginal Integrity of Class 2 Composite Restorations

Standardized, box-shaped approximal-occlusal cavities were prepared in 24 extracted human molars. All cavity margins were located within the enamel; gingival margins of the approximal box ended 1 mm above the cementoenamel junction. The cavosurface margins were beveled at a width of 0.5 mm with a diamond finisher. After preparation, the teeth were randomly assigned to four experimental groups (I-IV) containing six teeth each. Enamel treatment and application of bonding agents in Groups I through IV were performed according to the protocol summarized in Table 2, strictly following the manufacturers' directions (Table 1). Subsequently, the pre-treated cavities in Groups I through IV were restored with the fine-particle hybrid composites listed in Table 2. In approximal areas three composite increments and in occlusal areas two increments were applied. Each composite layer was light cured for 40 seconds (Translux EC; Kulzer). Removing excesses, contouring, and final finishing of the restorations were performed with diamond finishers (Composhape H40/H15; Intensiv, Lugano, Switzerland) and with flexible disks of decreasing grain sizes (Sof-Lex Pop-On, 3M Dental Products, St Paul, MN 55144).

Table 3. Enamel-Composite Bond Strength Measured in Groups I - IV

Group	Enamel Bonding Agent	Shear Strength (MPa)
I	Clearfil Liner Bond 2	24.2 ± 3.0
II	Etch & Prime 3.0	21.9 ± 1.4
III	Resulcin AquaPrime + MonoBond	34.0 ± 3.6
IV	Ecusit-Mono	26.3 ± 1.8

Vertical lines indicate significant differences; $P < 0.05$.

After placement of the restorations, the teeth were subjected in sequence to thermocycling and occlusal loading. Thermocycling included 2500 cycles within a temperature range of 50 K for the duration of 60 seconds at a minimum temperature of 5 °C and a maximum of 55 °C. The occlusal load involved 500,000 cycles with a force of 100 N. A stamp made of Co-Cr-Mo-alloy (Remanium CD; Dentaurum, Pforzheim, Germany) and coated with composite served as antagonist during occlusal load.

Marginal adaptation of the restorations was analyzed by scanning electron microscopy performed on epoxy resin replica models (Stycast; Grace, Westerlo, Belgium) before and after in vitro load. Marginal adaptation of the fillings was evaluated in steps of 100 µm at a X320 magnification according to the following parameters: perfect margin (defined as a continuous transition between filling and enamel); marginal gap; overhang; and marginal irregularity (characterized as a noncontinuous yet gap-free transition between filling and enamel). The percentage distribution of the varying qualities of marginal adaptation in the four groups was calculated for the approximal restoration margins. Statistical analyses took place with the H-Test (according to Kruskal-Wallis) and the Mann-Whitney U-Test.

RESULTS

Shear Bond Strength

Results of shear bond strength measurement are summarized in Table 3. The application of Resulcin AquaPrime MonoBond resulted in the significantly highest shear bond strength (34.0 MPa). The application of Etch & Prime 3.0 resulted in significantly lower values of shear bond strength (21.9 MPa) as compared to Ecusit-Mono applied after enamel etching with phosphoric acid (26.3 MPa). However, shear bond strength values using Clearfil Liner Bond 2 (24.2 MPa) and Ecusit-Mono (26.3 MPa) showed no statistically significant differences.

Scanning Electron Microscopic Analysis of the Etched Enamel Pattern

The SEM micrographs of the etched surfaces are found in Figures 2-4. Even by macroscopical inspection of the etched surfaces, differences could be detected. The surfaces treated with Resulcin AquaPrime showed a chalky appearance, similar to the clinically observed enamel surface changes after phosphoric acid etching. In contrast, enamel surfaces treated with Etch & Prime 3.0 appeared inhomogeneously dull; whereas, surfaces etched with LB PRIMER liquid A and B were characterized by a silky sheen rather than a dull appearance. The SEM

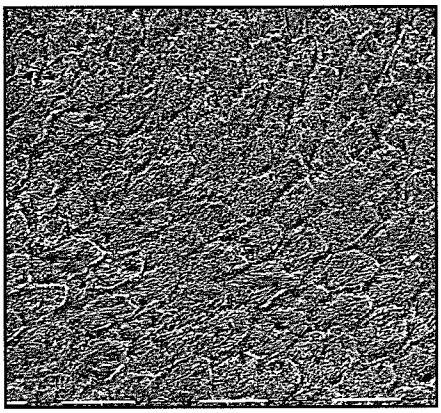

Figure 2. *Enamel surface after treatment with LB PRIMER liquid A and B (Group I) for a period of 30 seconds (original magnification X937.5; bar = 10 µm)*

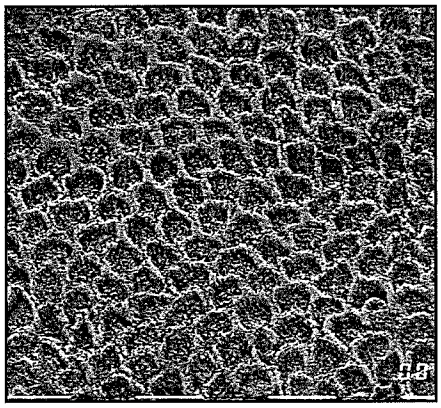

Figure 3. *Enamel surface after treatment with Etch & Prime 3.0 (Group II) for a period of 30 seconds (original magnification X937.5; bar = 10 µm)*

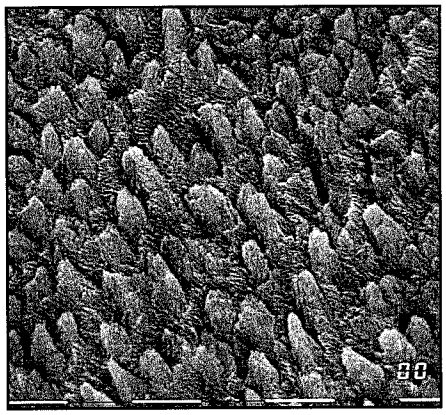

Figure 4. *Enamel surface after treatment with Resulcin AquaPrime (Group III) for a period of 30 seconds (original magnification X937.5; bar = µm)*

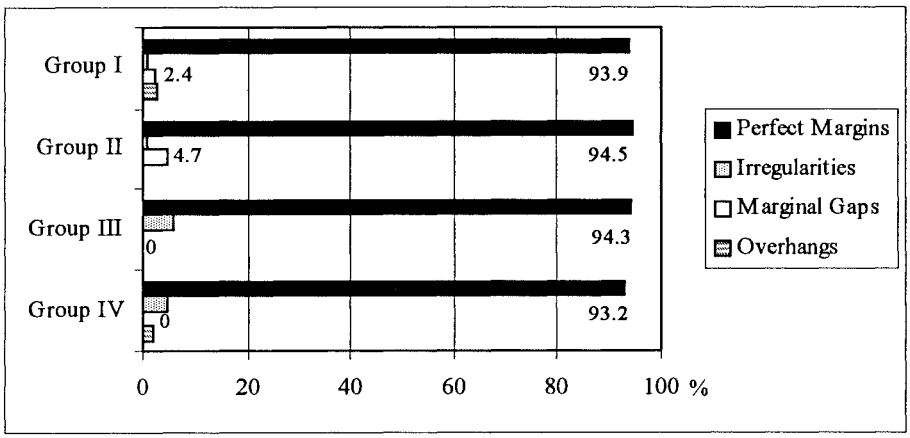

Figure 5. *Percentage frequency distribution of the different marginal quality criteria as detected on the approximal restoration margin after application of composite resin restorations in Groups I-IV (see Table 2)*

micrographs confirmed these observations by picturing the familiar etching pattern from phosphoric acid etching on the surfaces treated with Resulcin AquaPrime (Figure 4). Enamel surfaces treated with Etch & Prime 3.0 differed only slightly from this pattern (Figure 3); whereas, the enamel surfaces treated with LB PRIMER liquid A and B revealed only a relatively shallow etching effect (Figure 2).

Quantitative Marginal Analysis

The approximal, enamel margins of the restorations in Groups I to IV revealed high rates of perfect marginal adaptation after application of the fillings, reaching levels of over 93% (Figure 5). The differences between the groups with respect to the percentage frequency of perfect margins were not statistically significant ($P < 0.05$). The quality of the marginal adaptation decreased only slightly and insignificantly as a result of thermomechanical loading (Figure 6). Marginal gaps could be detected on 5.5- 6.0% of the analyzed marginal lengths in approximal areas after the thermomechanical load test.

DISCUSSION

It has been estimated that bond strengths of 17 to 20 MPa are required to resist contraction forces sufficiently to attain gap-free margins in resin composite restorations (Davidson, de Gee & Feilzer, 1984). Bond strengths of resin composites to phosphoric acid-etched enamel are typically in the range of 20 MPa (Gottlieb, Retief & Jamison, 1982; Munechika & others, 1984; Barkmeier, Shaffer & Gwinnett, 1986; O'Brien & others, 1987; Gilpatrick, Ross & Simonsen, 1991; Gwinnett & Kanca, 1992). Such bond strengths provide routinely successful retention of resin composites for a variety of clinical applications, including direct posterior composite restorations (Swift, Perdigao & Heymann, 1995). Resulcin AquaPrime MonoBond achieved the highest shear bond strength (34.0 MPa) among the self-etching products, as could be expected from its etching pattern. Surprisingly, Clearfil Liner Bond 2, in spite of causing the less distinct etching pattern, did not show significant differences in shear bond strength values (24.2 MPa) compared to Etch Prime 3.0 (21.9 MPa). A possible explanation for these findings may be found in the mode of failure caused by shearing forces. Resulcin AquaPrime MonoBond and Clearfil Liner Bond 2 break away primarily at the enamel bonding interface (adhesive failure), whereas Etch & Prime 3.0 fails to achieve sufficient adhesion in the bonding agent itself (cohesive failure). Furthermore, it was observed that layers of Etch and Prime 3.0 not covered with composite resin lost contact with the enamel just a few hours after water storage. However, negative effects on marginal integrity of resin composite restorations caused by the latter phenomenon could not be established in the present investigation.

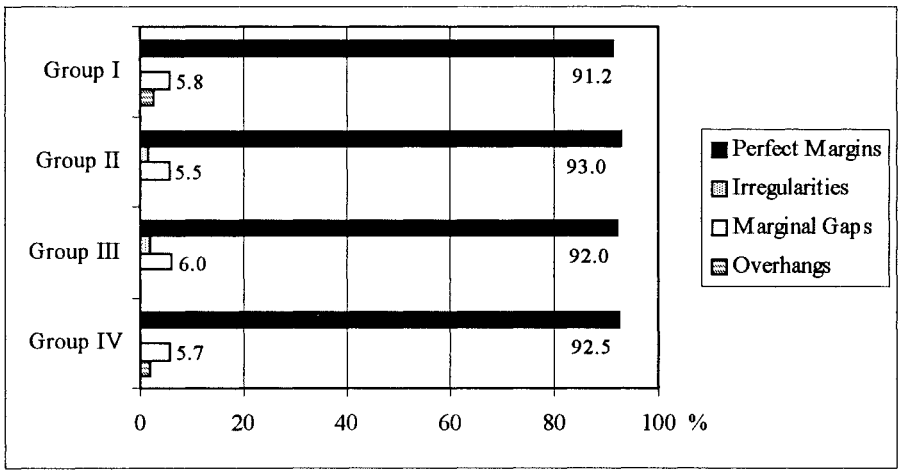

Figure 6. *Percentage frequency distribution of the different marginal quality criteria as detected on the approximal restoration margin after thermomechanical load of composite resin restorations in Groups I-IV (see Table 2)*

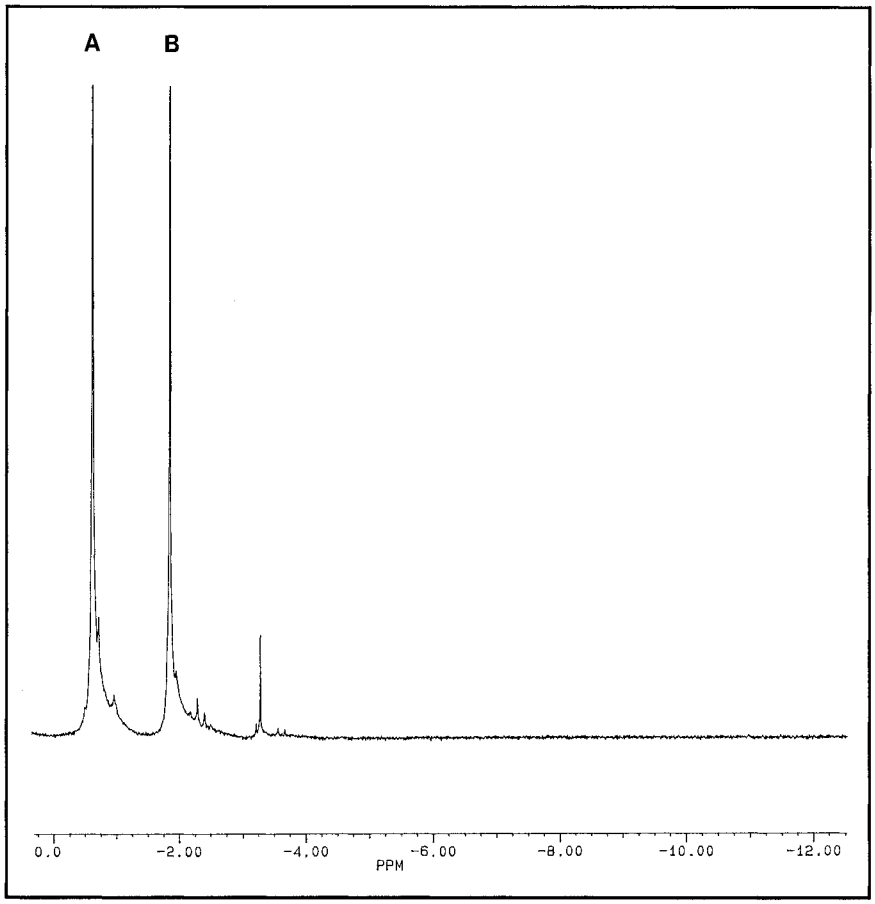

Figure 7. *^{31}P-NMR-spectrum of the mixture of phosphoric acid esters (in $CDCl_3$) contained in Resulcin AquaPrime. Peaks indicate 2-methacryloyloxyethyl-dihydrogenphosphate (a) and dimethacryloyloxyethyl-hydrogenphosphate (b).*

As clearly seen in SEM micrographs, primer materials investigated in Groups I through III had the capability to etch enamel. Since the primer is not rinsed after application, but air dried only, the calcium and phosphate ions that were dissolved from the hydroxyapatite crystals must be suspended in the watery solution of the primer. When the water is evaporated during air drying, the concentrations of solubilized calcium and phosphate within the primer may exceed the solubility product constants for a number of calcium phosphate salts (Yoshiyama & others, 1996). Presumably, minerals will then precipitate within the primer. These high concentrations of calcium and phosphate will tend to limit further dissolution of the apatite due to the common ion effects of calcium and phosphate (Yoshiyama & others, 1996) and thereby limit the depth of enamel surface demineralization. On the other hand, it is very likely that the binding of calcium ions to the phosphate residues in primer molecules contributes to the inactivation of the molecule's acidity. In addition, evaporation of water during air drying, as well as light curing of the primer and subsequently applied bonding agents, will restrict and inhibit the self-etching effect of the primer molecules.

Distinct differences in the enamel-etching pattern resulted from the application of self-etching primers to the enamel surface, depending on the particular product used. These differences are understandable in view of the specific esters contained in the self-etching primers. Resulcin AquaPrime and Etch & Prime 3.0 contain the considerably more acidic 2-methacryloyloxyethyl-dihydrogen-phosphate (Table 1; Figures 7 and 8), while LB PRIMER liquid A and B contain phenyl-hydrogen phosphate along with carbonic acid. Regarding Etch & Prime 3.0, it should be noted that contrary to the manufacturer's declaration, no pyrophosphoric acid ester could be detected, but rather exclusively its hydrolyzed components (Figure 8), which were much richer in 2-methacryloyl-oxyethyl-dihydrogen-phosphate than in dimethacryloyloxyethyl-monohydrogen-phosphate.

The interpretation of the SEM micrographs should take into consideration that the self-etching primers were removed from the enamel surface by an intensive rinsing with alcohol and acetone after the treatment times recommended by the manufacturer. Thus, it is conceivable that residues of the primer or possibly precipitates of calcium phosphates remained on the enamel surface and thereby masked the etching pattern. Consequently, the morphological evaluation of the etching effect of the various self-etching primers on the enamel surface was limited, depending on the specific methods chosen. However, a likewise shallow enamel etching pattern as observed in the present study for Clearfil Liner Bond 2 has also been reported by Perdigao & others (1997).

Due to its intrinsic acidity, the self-etching primer dissolves the enamel surface and thereby creates a three-dimensional microretentive surface pattern, while simultaneously promoting monomer infiltration. Depth of enamel demineralization and penetration depth of the bonding agent are therefore identical, since both processes run parallel to each other. As a result, light curing of these interpenetrated monomers and copolymerization with the overlying resin bonding agent and composite resin form a continuous bond with the enamel surface

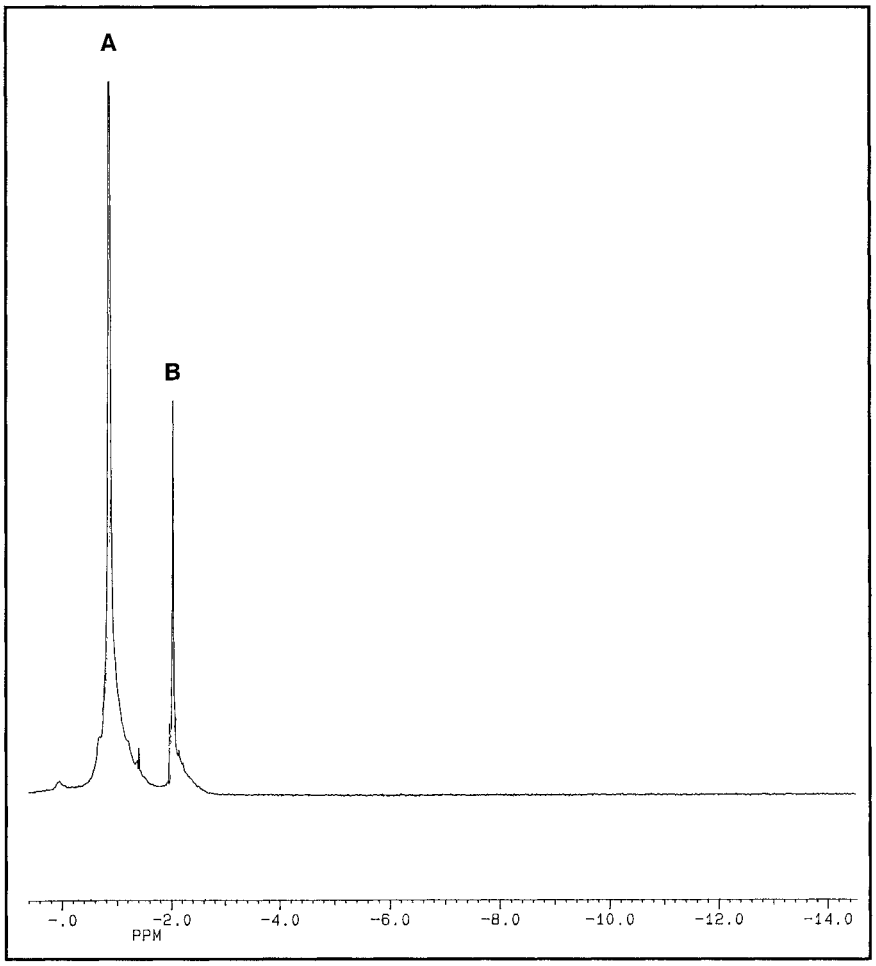

Figure 8. *^{31}P-NMR-spectrum of the mixture of phosphoric acid esters (in $CDCl_3$) contained in Etch & Prime 3.0. Peaks indicate 2-methacryloyloxyethyl-dihydrogenphosphate (a) and di-methacryloyloxyethyl-hydrogenphosphate (b); tetra-meth-acryloyloxyethyl-pyrophosphate could not be detected.*

capable of resisting the effect of microleakage (Schmit & Van Sint Jan, 1997). Comparison of the class 2 composite restorations in the four experimental groups of the present study did not detect any significant differences in marginal adaptation. After load, 91.2% - 93% of the approximal marginal length of the restorations showed a perfect marginal seal. The percentage of marginal gaps after thermomechanical load was only 5.5% - 6%. These results clearly documented that the use of self-etching primers in class 2 composite restorations can achieve a marginal integrity comparable to that attained by the conventional conditioning of the enamel with phosphoric acid. Marginal analysis with scanning electron microscopy was limited to the critical approximal sectors of the restorations because previous research had shown that load-induced marginal disintegration takes place especially in approximal areas of class 2 composite restorations (Bott & Hannig, 1995). Interpretation of the results should also bear in mind that in all groups the cavosurface margins were beveled at a width of ca 0.5 mm, as is generally required for class 2 cavities (Luescher & others, 1977; Porte & others, 1984; Ben-Amar, Metzger & Gontar, 1987; Hinoura, Sectos & Phillips, 1988; Dietschi & others, 1995). Beveling the cavity margins within the enamel enhances microretention and improves marginal adaptation (Luescher & others, 1977; Munechika & others, 1984; Moore & Vann, 1988). Class 2 composite restorations that were applied without beveling the approximal margins and subsequently subjected to an in vitro load test (under the identical conditions as in the present test) revealed gaps on 38.3% of the approximal restoration margins (Bott & Hannig, 1995).

New techniques should not be viewed as alternatives to well-established restorative methods unless they offer advantages from a scientific and clinical viewpoint. Compared to the conventional acid-etching technique, self-etching primers have the advantage of simplifying the application procedure. The products examined in this study are offered both as enamel and dentin bonding agents; thus, it is conceivable that a combined application of self-etching primers on enamel and dentin surfaces could take place without separate or selective acid etching. Further investigations are now in progress to examine the marginal adaptation of composite resin restorations applied with the use of self-etching primers in class 2 cavities with approximal margins located in dentin.

CONCLUSIONS

The results of this in vitro study indicated that self-etching primers can provide an effective alternative to conventional phosphoric acid etchants in conditioning the enamel surface to secure a durable bonding and marginal seal of composite resin restorations. The bond strength of the composite resin to bovine enamel attained by the tested self-etching primers was comparable to that attained by the conventional acid-etch technique, and was even stronger in the case of Resulcin AquaPrime MonoBond. Class 2 composite resin restorations placed using self-etching primers and those placed using the conventional phosphoric acid etchants did

not reveal any significant differences in marginal adaptation after thermomechanical loading. The self-etching primers evaluated in this study can be used on prepared enamel surfaces without phosphoric acid etching.

Acknowledgment

We would like to thank Mrs Astrid Röhl and Mr Axel Emmermann for their skillful assistance with the scanning electron microscopic analyses.

(Received 19 February 1998)

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Nanoleakage at the Dentin Adhesive Interface vs μ-Tensile Bond Strength

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Clinical Relevance
Overetching dentin with 35% phosphoric acid revealed no effect on short-term dentinal bond strength values but showed an increase in nanoleakage that raised concern about the long-term hydrolytic stability of resin and collagen fibrils in the resin-infiltrated dentin.

SUMMARY

Excessive etching of the dentin may decrease bond strength because the adhesive may fail to completely infiltrate to the base of the over-etched demineralized collagen network. The purpose of the present study was to evaluate the influence of increasing etching times on the microtensile bond strength of Single Bond and the leakage of silver ions within the hybrid layer. After etching occlusal dentin for 15, 30, or 60 seconds with 35% phosphoric acid gel, Single Bond was applied and cured for 10 seconds. Z100 was added and cured for 60 seconds. After 24 hours of water immersion, the teeth were sectioned into slices 0.7 mm thick, and hour-glass-shaped specimens were prepared. Alternate slices were either dried for 30 minutes in air, kept wet, or they were coated with fingernail varnish except for 0.5 mm around the bonded area. Only the varnished samples were then stained with 50% $AgNO_3$. Microtensile bond strength was tested using a Vitrodyne V-1000 universal tester. The samples of the stained group were embedded in self-curing PMMA and polished. All samples were observed with an SEM. Nanoleakage of silver ions was measured by exposure to laser ablation with an inductively connected plasma mass spectrometer and by electron dispersive elemental analysis. Increasing etching times seemed to have a negligible effect on bond strength of Single Bond, producing an average value of ca 38 MPa. However, the silver uptake increased upon prolonged

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Table 1. Composition of Materials Used in the Present Study

Material	Acid	Bonding Resin
Single Bond	(Etchant): 35% PA	HEMA BIS-GMA polyalkenoate copolymer photo initiators stabilizers ethanol
Z100		BIS-GMA TEGDMA photo initiator stabilizer inorganic filler (66 vol %): ZrO/SiO_2

HEMA = 2-hydroxyethylmethacrylate; BIS-GMA = bisphenol A glycidylmethacrylate; TEGDMA = tetraethylenglycoldimethacrylate.

etching times. Short-term results suggest that overetching has no detrimental effect on bond strength values of Single Bond. However, increased silver uptake, depending on the etching time, raises concern about the long-term stability of the bond.

INTRODUCTION

Acid etching enamel and dentin with the same high-concentration acid gel (40% phosphoric acid) followed by rinsing and air drying the cavity was first introduced by Fusayama and others (1979) to simplify the bonding process. It is known today, however, that air drying the demineralized collagen network may cause a collapse of the collagen network (Inokoshi & others, 1990; Sugizaki, 1991; ten Cate & others, 1991; Arends & Ruben, 1995; Carvalho & others, 1996a,b), leading to low bond strength values. Leaving the dentin visibly moist after acid etching and rinsing to prevent the collapse of the collagen fibril network has been shown to improve bond strengths (Kanca, 1992a,b; Gwinnett, 1992, 1994) and is now known as the "moist bonding technique." In the early 1990s most manufacturers provided acid gels with reduced concentration (ca 10%) for etching the dentin, since the procedure described by Fusayama and others (1979) using 40% phosphoric acid gel may have overetched the dentin. Now that the moist bonding technique is well established, 32-40% phosphoric acid gel, originally used on enamel only (Buonocore, 1955), is recommended for application on dentin, to further reduce the steps that are necessary to achieve good adhesion on dentin as well as on enamel. However, higher-concentration acid gels tend to dissolve the inorganic part of dentin (primarily hydroxyapatite crystals) more deeply. Research has shown that the dissolution of the hydroxyapatite can reach as deep as 10 to 12 µm into the superficial dentin and may prevent the subsequently applied primer and adhesive resin from reaching the bottom of the demineralized dentinal network (Van Meerbeek & others, 1993b) despite using a moist bonding technique. Therefore, application times of 5 to 15 seconds have been recommended to prevent overetching the dentin if 32-40% phosphoric acid gel is used.

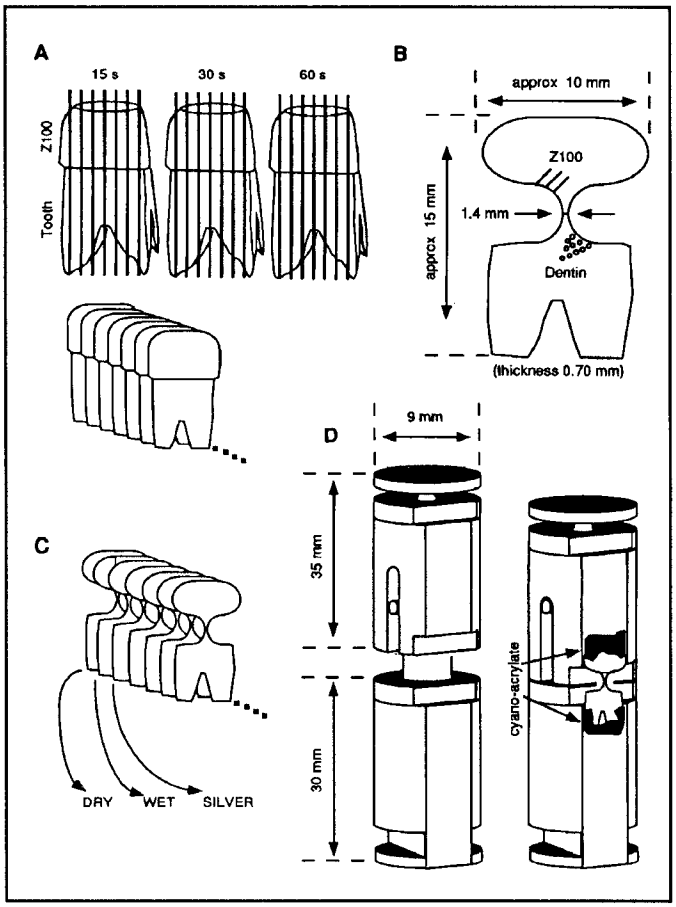

Figure 1. *A: Human third molars were used for each group depending on the etching time. B: Each tooth was sectioned into slices of approximately 0.7 mm thickness. Each slice was reduced from the mesial and distal aspects at the resin-dentin interface into an hour-glass shape. C: Alternate slices of each tooth were assigned to either dry or wet storage or were submitted to staining with silver nitrate after covering the tooth with varnish except for 0.5 mm around the bonded area. D: The hour-glass-shaped slices were attached to a Ciucchi device with cyanoacrylate and tested in tension until failure to determine tensile bond strength.*

Table 2. Microtensile Bond Strength Values (MPa) for Specimens That Were Etched for 15 Seconds, 30 Seconds, or 60 Seconds with Dry or Wet Storage or Stained

Etching Time	15 Seconds	30 Seconds	60 Seconds
Dry	41.51 ± 12.93 (7)	35.25 ± 16.17 (7)	44.87 ± 17.87 (7)
Wet	40.19 ± 9.66 (6)	27.90 ± 16.65 (7)	25.70 ± 7.36 (6)
Stained	39.34 ± 16.30 (6)	35.04 ± 15.20 (6)	37.06 ± 14.03 (7)

Values are x ± SD (number of slices per group). Groups connected by vertical lines are not significantly different ($P > 0.05$).

The purpose of the present study was to examine the effect of various etching times on the bond strength of Single Bond using 35% phosphoric acid gel and the association of bond strength with leakage of silver ions within microscopic spaces of the hybrid layer. The hypothesis to be tested was that increasing etching times would result in lower bond strength and higher content of silver ions in the hybrid layer.

METHODS AND MATERIALS

Nine extracted human third molars stored at 4 °C in isotonic saline containing 0.2 % sodium azide were used in the present study.

The occlusal enamel was reduced perpendicularly to the long axis of the teeth by grinding them with wet aluminum oxide paper (240- and 600-grit; Carbimet Paper Discs, Buehler Ltd, Lake Bluff, IL 60044) until no more enamel was visible at X5 magnification.

All bonding procedures were done using Single Bond (Lot 19970204, Exp Date 02/2000; 3M Dental Products, St Paul, MN 55144), including a 35% (wt) phosphoric acid gel and Z100 restorative resin composite (Lot 19970121, Exp Date 10/1999; 3M Dental Products) (Table 1).

The teeth were randomly assigned to three groups according to the etching time (Group 1: 15 seconds, Group 2: 30 seconds, Group 3: 60 seconds; Figure 1A). After acid etching, the teeth were rinsed with water for 10 seconds and blot dried (Kimwipes; Kimberly-Clark, Roswell, GA 30076), leaving the dentin moist as per the manufacturer's instructions. Two consecutive coats of Single Bond were applied with a fully saturated brush. The surface was gently dried for 5 seconds (oil-free Fisherbrand Precision Duster; 1,1,1,2-tetrafluoroethane & methyl oxide; Fisher Scientific, Pittsburgh, PA 15275) and light cured for 10 seconds (Optilux 500; Demetron/Kerr, Danbury, CT 06810; 870 mW/cm^2). Two layers of Z100 resin composite were added to the bonded dentinal surface, each 1.5 mm in thickness, and each layer was light cured for 60 seconds with the same Optilux 500. After immersion in water for 24 hours, the teeth were sectioned in a buccolingual direction into slices approximately 0.7 mm thick (Figure 1A) with an Isomet saw (Buehler). The exact thickness of each specimen was measured with an electronic caliper (Model CD-6BS; Mitutoyo, Tokyo, Japan). To obtain a cross-sectional surface area of approximately 1 mm^2 for the bond strength measurement, the adhesive dentin interface was reduced from the mesial and distal aspects to an average width of 1.4 mm with an air-rotor and a fine-grain diamond, resulting in hour-glass-shaped specimens (Figure 1B). Due to concern that these small specimens might dry out and give higher than normal bond strength values, both wet and dry specimens were tested. Concern that conversion of $AgNO_3$ to metallic grains of silver might stress the resin-dentin bond dictated a third group for comparison. Alternate slices were either dried for 30 minutes in air (Dry), kept wet (Wet), or they were completely coated with fingernail varnish except for 0.5 mm around the bonded area (Figure 1C). Only the varnished samples (n = 19, Table 2) were then immersed in 50% $AgNO_3$ for 1 hour prior to immersion into a freshly prepared Kodak photo-developer (Dektol, Cat 146 4726; Eastman Kodak, Rochester, NY 14650) for 12 hours (Stained). All specimens were consecutively bonded to a Ciucchi device (Figure 1D) using cyanoacrylate (Zapit; DVA,

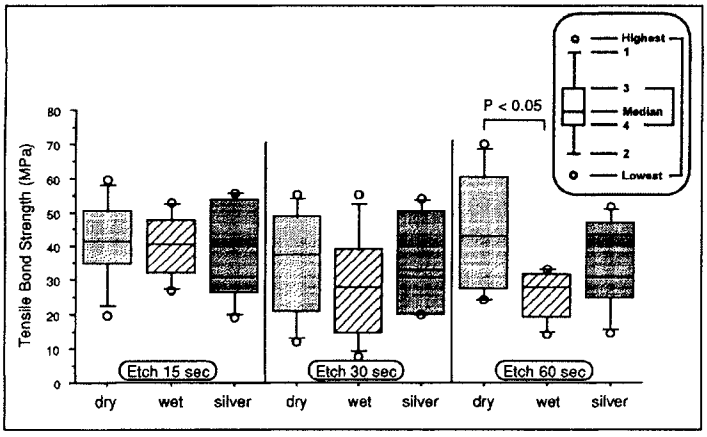

Figure 2. *Boxplot of the resulting microtensile bond strength values according to the etching time and depending on the storage type (dry, wet, or silver). There were no significant differences found in μ-TBS among the different etching times or among the testing conditions. Insert shows the distribution of values in a boxplot drawing. 80% of the values lie between 1 and 2, 50% between 3 and 4. Median: 50% of values lie above, the other 50% below this mark. H = highest; M = median; L = lowest.*

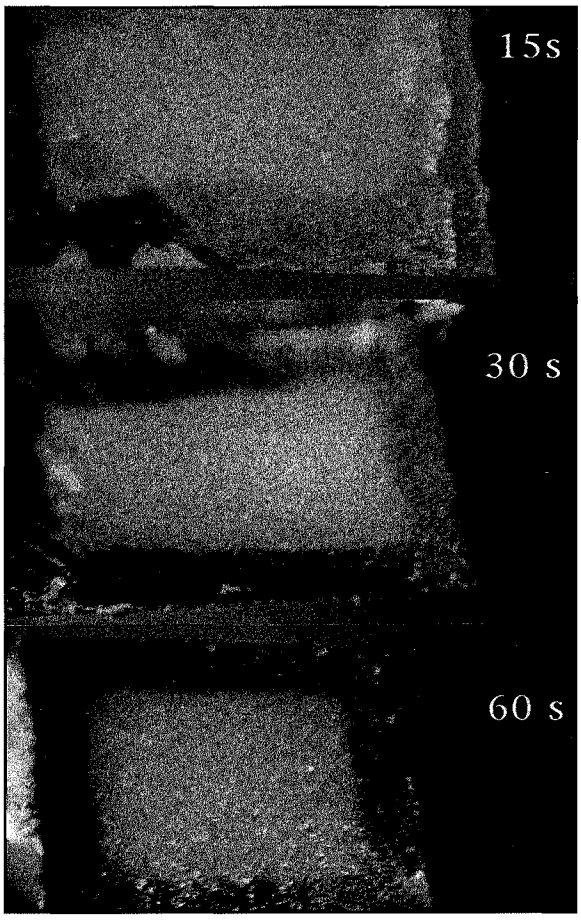

Figure 3. *Light microscopic evaluation of the approximately 1 x 1 mm fracture sites of the silver group depending on the etching time (15 seconds, 30 seconds, or 60 seconds). All samples showed a dark circumferential band due to silver penetration and clear center portions. Most fractures were adhesive on top of the hybrid layer with varying degrees of adhesive resin remnants remaining. (magnification X20)*

Corona, CA 91720). This device ensures that pure tensile forces are applied to the specimens. The specimens were tested for microtensile bond strength (μ-TBS) using a 5 kg load cell in a Vitrodyne V-1000 universal tester (Chatillon, Greensboro, NC 27409) that was operated by a computer using Material Witness Software 2.02 (Chatillon).

After measuring shear bond strength, samples of the dry and of the wet group were sputter coated (Model K 550; Anatech LTD, Alexandria, VA 22309) with a 10 nm-thick layer of gold prior to observation with a Phillips XL-30 SEM (Phillips Instruments Co, Norcross, GA 30071) using an accelerating voltage of 25 keV. Representative photographs (Polaroid, Model 545; 4x5 Land Film, Type 55 Pos/Neg; Polaroid Corp, Cambridge, MA 02139) of the fracture sites were taken.

The dentinal fracture sites of the silver group specimens were photographed with a Nikon F3 (Ektachrome 64 T; Nikon Inc, Melville, NY 11747) attached to a Nikon SMZ-U light microscope at X20 magnification. After embedding these specimens with self-curing PMMA (Quickmount; Fulton, Saxonburg, PA 16056), the dentin surface was polished using hand pressure first on stationary wet aluminum oxide paper (320-, 400-, 600-grit; Carbimet Paper Discs, Buehler), and then with diamond polishing pastes (N° 15, 10-20 μm; N° 6, 4-8 μm; N°1, 0-2 μm; Kay, Deerfield Beach, FL 33442) diluted with a water-soluble lubricant (Thinner Lubricator; Kay) on nylon polishing cloths (Buehler) that were mounted on stationary glass slabs (diameter: 7.3 cm). Finally, 0.25 μm polishing spray (N° 100,000; Kay) was used on microcloth polishing cloths (N° 40-7212; Buehler). The samples of the stained group were sputter coated with 10 nm-thick carbon, and one tooth slice per group (15-, 30-, and 60-second etching time) was exposed to laser ablation with an inductively coupled plasma mass spectrometer (ICPMS; Georgia State University, Atlanta, GA 30303). Backscattered SEM images were taken from all samples of the stained group with a Phillips XL-30 scanning electron microscope, and an electron dispersive element analysis was performed with an EDAX attached to the SEM to confirm the ICPMS data. The EDAX values were measured throughout in three consecutive windows of 4 μm x 4 μm moving vertically over a cross section of the specimen from top to bottom.

Experimental Design and Statistical Analysis

Three etching times were used: 15, 30, and 60 seconds. Three teeth were included in each group. After bonding, construction of resin composite build-ups, and storage in water for 24 hours, each tooth was serially sectioned longitudinally into seven 0.7 mm-thick slabs. The seven slabs of each of the three teeth in each group were pooled (21 slabs) and randomly assigned to the wet, dry, or stained subgroups. Thus, each group contained 21 slabs. There were three groups of 21 slabs, for a total of 63 slabs. One slab was lost during processing, and one slab from each group broke during trimming to an hour-glass configuration, leaving 59 slabs for testing.

RESULTS

Bond strength values (μ-TBS) of Single Bond/Z100 are summarized in Table 2. Overall, the μ-TBS showed moderate deviation from an average value of approximately 38 MPa (Table 2). There were no significant differences found in μ-TBS among the different etching times ($P = 0.270$) or among the testing conditions ($P = 0.138$). There was no interaction between the two main variables ($P = 0.605$). Figure 2 shows the results of the μ-TBS measurements graphically.

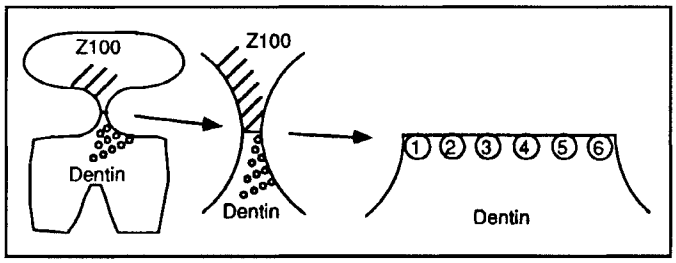

Figure 4A. *Laser ablation sites along the dentin just below the bonded surfaces. The silver nitrate-stained samples used for the inductively coupled plasma mass spectrometry were submitted to laser ablation at six locations close to the hybrid layer.*

The light microscopic pictures of the silver group (X20 magnification) showed silver staining at the edges but clear center portions of the approximately 1 mm²-wide fracture sites. Most fractures were adhesive on top of the hybrid layer with varying amounts of bonding resin fragments remaining (20% to 70%; Figure 3). Only very few fractures included partly cohesive failures in the resin composite, with remnants of the resin composite adhering to the top of the hybrid layer.

Table 3. Data from the ICPMS for the Concentration of Calcium (Ca^{44}) and Silver (Ag^{107} and Ag^{109}) Depending on the Etching Time

Location	Elements	Etching Time (Seconds)		
		15	30	60
1	Ca^{44}	0.72*	2.05	0.83
	Ag^{107}	2.94	51.25	--
	Ag^{109}	2.42	46.19	--
2	Ca^{44}	0.93	1.68	0.86
	Ag^{107}	0.38	29.93	--
	Ag^{109}	0.31	20.70	--
3	Ca^{44}	0.85	1.67	0.81
	Ag^{107}	0.22	22.56	--
	Ag^{109}	0.20	11.90	--
4	Ca^{44}	0.87	1.16	0.86
	Ag^{107}	0.26	16.70	--
	Ag^{109}	0.22	10.21	--
5	Ca^{44}	0.86	1.60	0.74
	Ag^{107}	0.45	24.83	--
	Ag^{109}	0.41	17.81	--
6	Ca^{44}	0.82	2.42	0.89
	Ag^{107}	4.81	48.25	17.58
	Ag^{109}	5.47	29.70	14.15

*Values are counts/24 sec x 10^6

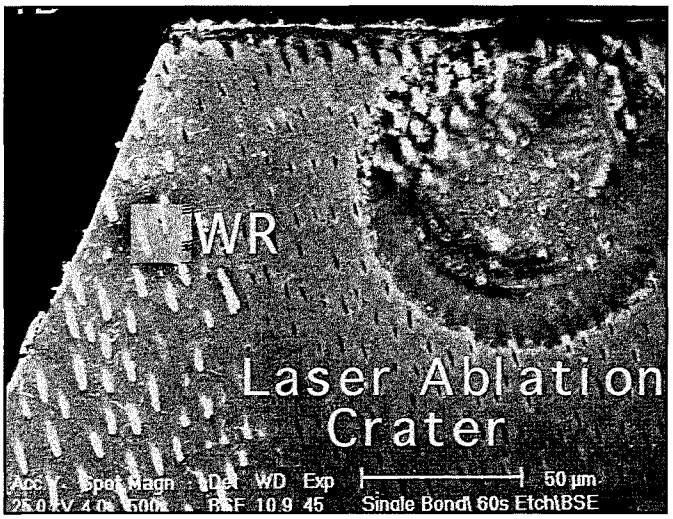

Figure 4B. *SEM picture of a laser ablation crater after inductively coupled plasma mass spectrometry. The whitish rectangle shows a respective window (16 µm²) that was used for the subsequent electron dispersive elemental analysis (EDAX). (magnification X470)*

In contrast to the relatively constant µ-TBS values, the ICPMS results (Figures 4A, 4B) identifying the amount of calcium and silver (Table 3) beneath the debonded surface showed a five- to eightyfold increase in silver for the sample that was etched for 30 seconds, compared to the sample with a 15-second etching time. The sample that was etched for 60 seconds did not reveal high amounts of silver, which was very probably the result of too extensive polishing (Table 3) that may have inadvertently removed the silver. The EDAX analysis, however, revealed increasing amounts of silver depending on the etching time. The sample with 15-second etching time showed an average silver ion content of 3.10, 3.17, and 1.10 atomic % (according to the three consecutive windows of 16 µm² used for measurement; Figure 5A), while the hybrid layer had a thickness of 2.5 µm. Increasing the etching time to 30 seconds increased the silver content only slightly (2.88, 3.24, and 0.85 atomic % respectively) but resulted in a hybrid layer that was two times as thick (5 µm; Figure 5B). Finally, 60-second etching times resulted in a considerable increase of the silver content (6.89, 2.39, and 1.55 atomic %) in a hybrid layer that was up to 10 µm thick (Figure 5C). These results are summarized in Table 4.

DISCUSSION

In order to obtain proper adhesion of resinous materials to dentin, it is essential to create a hybrid layer at the resin-dentin interface (Nakabayashi,

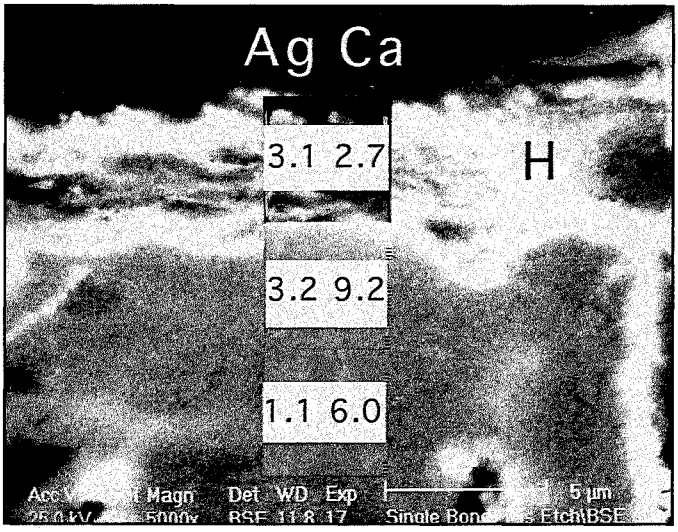

Figure 5A. *EDAX of the silver-stained specimens to determine the silver content at their carbon-coated surface. After 15 seconds of etching an average silver content of 3.10, 3.17, and 1.10 atomic % was detected in the three consecutive 4 x 4 µm windows used for the measurement. The width of the hybrid layer (H) was approximately 2.5 µm. (magnification X4700)*

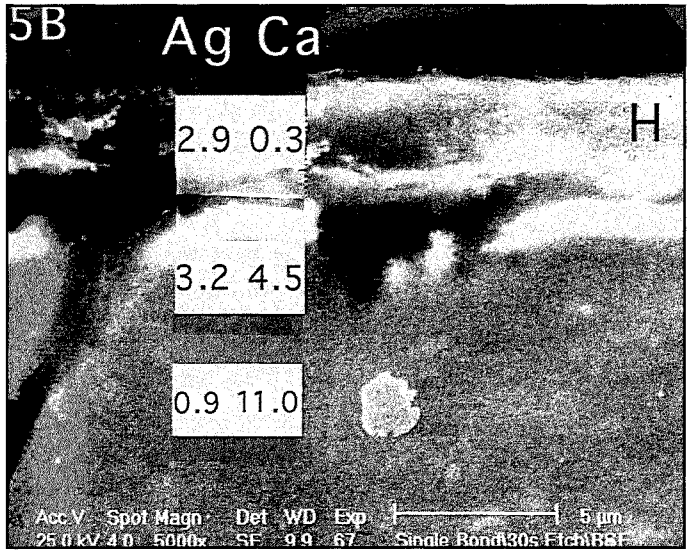

Figure 5B. *After 30 seconds of etching the average silver content was 2.88, 3.24, and 0.85 atomic % respectively with a hybrid layer (H) that was 5 µm thick. (magnification X4700)*

1982; Eick & others, 1991, 1992, 1993a,b; Gwinnett & Kanca, 1992, Van Meerbeek & others, 1992, 1993a,b; Pashley & others, 1993, Walshaw & McComb, 1994). The hybrid layer is created by the penetration and polymerization of adhesive monomers after removal and/or modification of the smear layer and superficial demineralization of the dentin. Ideally, adhesives should fully penetrate to the bottom of the demineralized zone and fill any voids caused by the demineralization process, to prevent any microleakage. However, according to Sano and others (1994b), who used electron-dense silver nitrate as a tracer to identify regions of demineralized dentin that were not infiltrated with resin, concerns remain as to the extent dentinal adhesives really are able to completely fill the demineralized collagen fibril network. Silver nitrate is an excellent tracer, as the silver ions are very small, and, due to the high solubility of silver, highly concentrated solutions can be prepared. Recent research data on the hybrid layer formation by various dentin bonding systems using SEM and TEM and the same tracer (Sano & others, 1995a,b) indicated the presence of submicron spaces between resinous material and demineralized collagen fibers that allowed accumulations of silver. Leakage in these nanometer spaces (termed "nanoleakage") raises concern about the long-term hydrolytic stability of both the collagen and the adhesives in the hybrid layer. In the present study, the formation of an excessively thick zone of superficially demineralized dentin was enhanced by extending the etching time. Investigation of the 1 mm² fracture sites by light microscopy (Figure 3) revealed a circumferential band of dark stain that left the center portion clear, confirming a lateral permeation of silver ions, as hypothesized by Sano and others (1994b; 1995a,b). Although the silver ions were not able to penetrate through and through, they seemed to have undermined the dentin-adhesive interface via diffusing within the hybrid layer. Despite this finding, most of the bonds failed adhesively at the top of the hybrid layer.

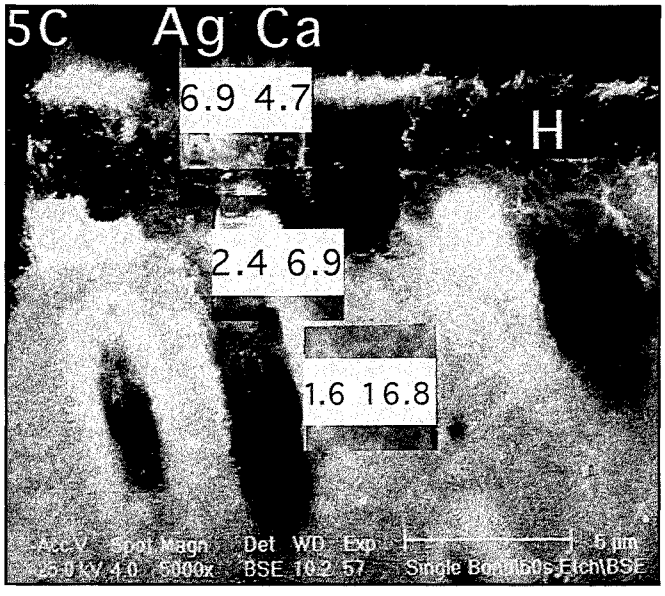

Figure 5C. *An etching time of 60 seconds resulted in a silver content of 6.89, 2.39, and 1.55 atomic % in the respective windows. The thickness of the hybrid layer (H) was ca 10 µm. (magnification X4800)*

Table 4. EDAX Analysis for Ag and Ca in Dentin Specimens Etched with 35% Phosphoric Acid

Etching Time	Hybrid Layer Thickness	Location	Ag (atomic %)	Ca (atomic %)
15 seconds	2.5 μm	top of hybrid	3.10	2.66
		bottom of hybrid	3.17	9.18
		mineralized dentin	1.10	15.96
30 seconds	5.0 μm	top of hybrid	2.88	0.32
		bottom of hybrid	3.24	4.50
		mineralized dentin	0.85	10.56
60 seconds	10.0 μm	top of hybrid	6.89	4.66
		bottom of hybrid	2.39	6.85
		mineralized dentin	1.55	16.78

Elemental dispersive spectrometry detects silver only on the surface of a carbon-coated sample. To obtain data on the concentration of silver ions in deeper portions of the stained specimens, we used laser ablation that was inductively coupled to a plasma mass spectrometer (ICPMS). As shown in Table 3, the data revealed a considerable increase of silver content if the etching time was extended to 30 seconds. In addition, the silver concentration was higher at the lateral ablation sites (positions 1 and 6, Table 3 and Figure 4A) than in the center ablation sites, which also supported the lateral permeation theory (Sano & others, 1994b, 1995a,b). However, the ICPMS data need to be interpreted carefully, since the smallest crater size that resulted in recordable spectrometric data was 70 μm in diameter and approximately 60 μm in depth (Figure 4B), whereas the thickness of the hybrid layer varied from only 2.5 μm (15-second etching time, Figure 5A) to 10 μm (60-second etching time, Figure 5C). Due to this discrepancy (i e, the laser craters would need to be reduced to less than the thickness of the hybrid layer to result in data that are truly related to nanoleakage), an EDAX analysis was performed to limit the obtained values for the silver concentration to the respective thickness of the hybrid layer. As shown in Figures 5A to 5C, electron dense bands of silver were visible at the top and at the bottom of the hybrid layer for all specimens. Whereas these bands were almost similar in thickness at the top of the hybrid layer, an increased thickness could be detected for the 30-second and the 60-second samples, suggesting that extensive overetching of the dentin resulted in a pronounced failure of the adhesive to fully infiltrate the demineralized zone. Diffusion time may have been too short for monomers to diffuse that deeply. Alternatively, dilution with water, which is needed to expand the demineralized collagen network, may have competed with resin for the surface of the collagen fibrils. However, the microtensile bond strength (μ-TBS) data (Table 2) were reasonably high for all specimens (ca 38 MPa) and surprisingly failed to reveal significant differences (Figure 2). It may be hypothesized that the observed primarily adhesive failures of the bonds and the occurrence of a silver band at the top of the hybrid layer of all samples were due to a lack of intimate contact between the composite resin (Z100) and the one-bottle bond (Single Bond). This would imply that the bond between the one-bottle adhesive and the resin composite was tested rather than the bond of the adhesive layer to dentin, which would further imply that the bond to dentin exceeded the measured values (since no cohesive failures in dentin were observed). However, the nonoccurrence of cohesive failures in dentin may only be true for short-term measurements when the ultimate tensile strength (UTS) of the partly resin-infiltrated collagen fibers (UTS of demineralized human dentin: ~ 30 MPa, Sano & others, 1994a) exceeds the tensile forces applied to measure dentinal bond strength of adhesives. After slow hydrolytic degradation of the resin and the collagen fibers in the submicron spaces of the hybrid layer, different results may be obtained. More research is indicated on degradational processes within the submicron spaces of the hybrid layer. Other adhesive systems should be evaluated using the same methodology.

CONCLUSION

Within the limits described above, the present study failed to confirm the hypothesized dependence of dentinal bond strength values on etching times. However, an increase in leakage of silver ions could be shown with increasing etching time. Studies on the effect of long-term water storage on similarly overetched specimens may reveal information on the hydrolytic stability of resin and collagen fibers in the resin-infiltrated dentin.

Acknowledgments

This work was supported, in part, by grant DE 06427 from the National Institute of Dental Research and by the Biocompatibility Group at the Medical College of Georgia.

(Received 11 March 1998)

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Award of Excellence

I am pleased and honored to present this award to José Medina on behalf of the Academy of Operative Dentistry. I will attempt here to distill almost a half century of his service to dentistry into a brief summary.

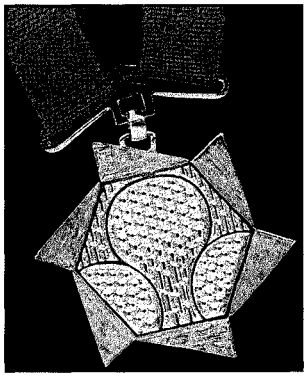
José is a native of Puerto Rico and received his early formative education there. He then attended Johns Hopkins University in Baltimore, before entering The Baltimore College of Dental Surgery at the University of Maryland in 1944. He graduated cum laude in 1948 with a DDS degree and was appointed to the faculty. José progressed up the academic ladder to professor and department head in 1961, and was appointed assistant dean in 1964. In 1967 the Medical Center at the University of Florida in Gainesville was becoming a reality, and José accepted the position of

José Medina

associate dean and professor of clinical dentistry. In 1969 he became dean of the College of Dentistry at Florida, an office he held until 1974.

José developed into a renowned and esteemed lecturer. He has the ability to present his subject within a clear and concise framework. Consequently, over the years he has given approximately 450 registered chair table television clinics and formal papers in Spanish and in English. These were presented in 22 states, Puerto Rico, Central America, Mexico, the Bahamas, Canada, South America, Germany, and Spain. In 1991 Chile presented to José the award of Knight Commander of the Order of Bernardo O'Higgins at an impressive ceremony at the Ministry of Health in Santiago.

The study club concept is unique to dentistry and is most valuable in teaching and learning operative skills. At one point José was mentor to clubs in New England; Central Florida; the US Naval Dental School Gold Foil Seminar at Bethesda, Maryland; and the Hollenback Seminar. His interest in gold foil led José to the Academy of Gold Foil Operators, still an active organization, and the forerunner of this academy. He served as editor of its journal until becoming president in 1965. He received that Academy's Distinguished Member Award in 1986. Dr Medina also served as the president of the Academy of Operative Dentistry in 1991, and has made numerous contributions to the academy's current status. He was awarded the Hollenback Prize in 1985.



In his long and distinguished career, José has never forgotten his dedication to the private practitioner. His sensitivity to the quality of treatment rendered in the individual dental office has always been foremost in his teaching methods. The inspiration and talent he displays is a guide to those of us who are fortunate to know him. His primary focus is the pursuit of excellence in delivering health care to our patients. It is therefore with great personal honor that I present the Award of Excellence on behalf of this academy to Dr José Medina.

PAUL H LOFLIN

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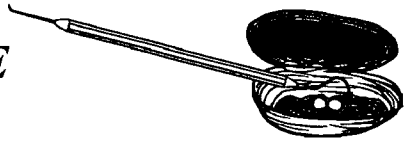

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E-Z GOLD TO RESTORE CONTACT IN A PORCELAIN CROWN (BRIDGE)

Contributed by:
Dr Lloyd Baum, Loma Linda, CA

An aggravation that frequently confronts the clinician is discovering that a beautiful, well-fitting porcelain crown is deficient in approximal contact. Cementation with a negative contact is considered unacceptable. Yet having the patient return for another appointment and returning the case to the lab for porcelain application is not a popular choice either. A third option is available: rebuild the contact point with E-Z Gold and cement the crown at the same appointment.

This process is simple and easy to accomplish by preparing an elliptical type of cavity at the contact area: e g, a round diamond stone, size #6, is quite adequate. The cavity need be only 1 - 1.5 mm deep. Peripheral retention is established with an inverted cone carbide bur (#35) (Figure 1). The porcelain crown may be hand-held during preparation of the cavity in the porcelain. Application of the gold, however, requires bench-top stability. This is readily achieved by embedding the crown on its side within some soft compound. The cavity is now facing upward on the laboratory bench and is ready to be filled with gold.

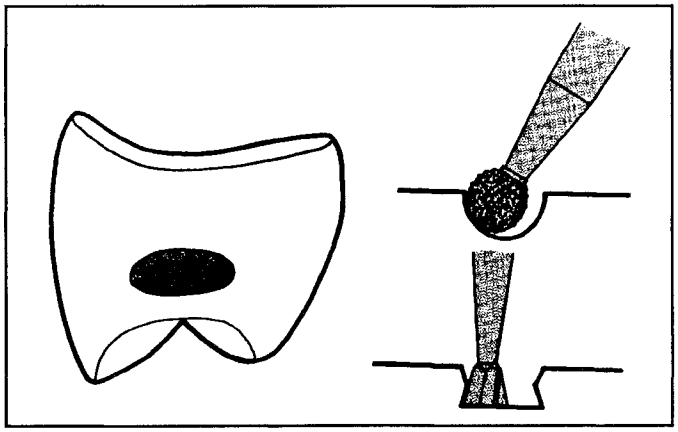

Figure 1. *Contact area cavity preparation using a diamond stone and inverted cone carbide bur*

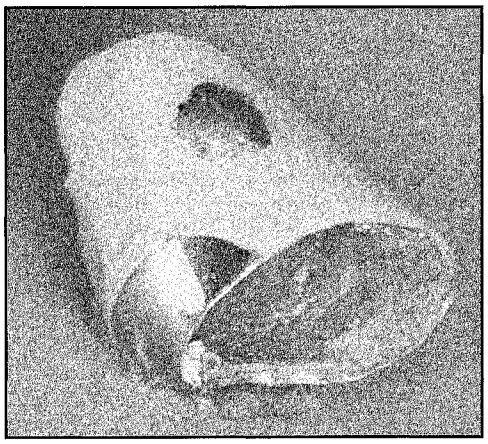

Figure 2. *Completed E-Z Gold restoration*

Cutting a cavity without chipping the porcelain is not hazardous if the cutting is done slowly with water coolant. The round diamond stone establishes the size, shape, and depth of the cut; the inverted cone flattens the floor and provides the retention.

E-Z Gold is then compacted into the cavity with gold foil pluggers and overbuilt in contour to achieve sufficient positive bulk contact. Burnishing and work-hardening the surface of the gold is done with a discoid carver or a file (Figure 2). After the surface is hard and free from porosity, the crown is broken free from the compound and is tried in the mouth. Carbon paper between the crown and the adjacent tooth will mark excess contact spots, which can be removed with a fine garnet or cuttle disk. When properly fitted, the crown is polished and permanently cemented.

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MAY-JUNE 1999 • VOLUME 24 • NUMBER 3 • 129-192

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