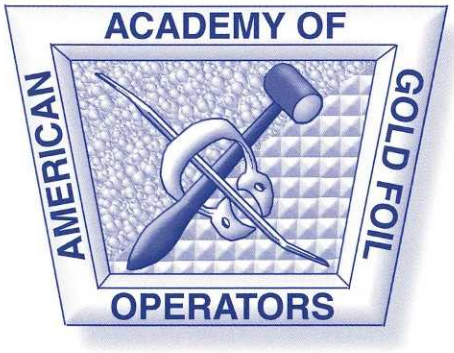
OPERATIVE DENTISTRY



may-june 2000 • volume 25 • number 3 • 145-248

(ISSN 0361-7734)

OPERATIVE DENTISTRY

MAY-JUNE 2000 • VOLUME 25 • NUMBER 3 • 145-248

Aim and Scope

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 are also 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 *Operative Dentistry,* Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186. Periodicals postage paid at Indianapolis, IN, and additional mailing offices. **Postmaster:** Send address changes to: *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186.

Subscriptions: Fax (317) 852-3162

Yearly subscription in USA is $60.00; all other countries, $70.00 (sent by air where appropriate); dental students (send verification of student status), $25.00 in USA; other countries, $34.00; single copy in USA, $14.00; other countries, $17.00. For back issue prices, write the journal office for quotations. Make remittances payable (in US dollars only) to *Operative Dentistry* and send to the above address. Credit card payment (Visa, MasterCard, or JCB—Japanese equivalent) is also accepted by providing card type, card number, expiration date, and name as it appears on the card.

Contributions

Contributors should study the instructions for their guidance printed in this journal and should follow them carefully.

Permission

For permission to reproduce material from *Operative Dentistry* please apply to *Operative Dentistry* at the above address.

The views expressed in *Operative Dentistry* do not necessarily represent those of the Academies or of the Editors.

Editorial Office

Operative Dentistry
Indiana University School of Dentistry, Room S411
1121 West Michigan Street, Indianapolis, IN 46202-5186
Telephone: (317) 278-4800, Fax: (317) 278-4900
URL: http://www.jopdent.org/

©Operative Dentistry, 2000, **25**, 145-146

In Memoriam

Miles R Markley

Miles R Markley, DDS, of Denver, Colorado, died on January 31, 2000, at the age of 96 of complications from a stroke. He conducted a private dental practice for 52 years, officially retiring in 1979. However, Miles never gave up thinking about and working in his profession. During his career, Dr Markley became a "dental superstar!"—he was a noted author, clinician, innovator, inventor, lecturer, teacher, and role model for healthy living. His effect on the clinical practice of dentistry in our profession is inestimable. In both his professional and personal life, Miles R Markley practiced what he preached. His goal was dental restorations that would last a lifetime, and much of his work withstood more than 60 years. He practiced preventive dentistry through diet, hygiene, and the saving of diseased and injured teeth through conservative restoration. He firmly believed that people need never lose their teeth.

Dr Markley was a chairside general dentist with a conscience. During his first five years in practice he noted with great concern that he was not producing durable restorations. As he studied his failures, he concluded that the GV Black-type of cavity preparations and the amalgam restorations that he had been taught to place in dental school could not provide the longevity that his patients deserved. To meet his more exacting requirements over his lifetime of practice, he:

- developed and refined conservative cavity preparations that preserved tooth structure.
- designed new dental burs and instrumentation for preparing these smaller restorations.
- perfected anatomical matrices, which were properly wedged for dental amalgams.
- emphasized the importance of the use of the rubber dam.
- perfected pin-retained amalgams and developed the first practical, commercial pin system.
- stressed that prevention (oral hygiene/nutrition) and operative dentistry MUST coexist.
- developed an exacting and accurate casting process for gold restorations.
- emphasized the importance of teaching patients how to maintain excellent oral hygiene.

Miles R Markley
1903-2000

It is interesting to note that Dr Markley carried out his extensive clinical research without academic affiliation or outside funding. Dr Markley never ceased his quest for learning. Even in his later years, he could be seen in the front row at dental meetings taking notes and paying careful attention to the guest speaker.

Miles, a native of Juniata, Nebraska, was born at home on November 5, 1903. The son of a dentist, he attended the University of Nebraska for his predentistry education. He received his DDS from Denver University in 1927, and was awarded the diamond-studded Delta Sigma Delta pin for graduating with the highest honors. Following his graduation, he practiced with his father in Kimball, Nebraska, for nearly six years. He and his wife, Winnifred, then moved to Denver at the bottom of the Depression in 1932. From his earliest years of practice, Miles was an avid stu-

dent of his profession. He directed the Mile Hi Study Club in Denver for 15 years; and commuted to Nebraska for 23 years to lead the Panhandle Region Study Club (renamed the Miles Markley Panhandle Study Club in 1977). In addition, Dr Markley was past president of the Denver and Colorado Dental Societies, and the Colorado State Board of Dental Examiners.

For 37 years (1946-1983), Dr Markley served as a civilian consultant in restorative dentistry to the surgeons general of the US Army, Navy, and Air Force (Department of Defense). In this capacity he traveled throughout the United States and overseas, where he conducted state-of-the-art courses in restorative dentistry and fixed prosthodontics for military dental officers. Dr Markley often served in this capacity at considerable personal sacrifice and expense. In the early 1960s, during his first lecture trip to the Philippines, Hawaii, and Japan, he traveled at night and lectured during the day. Over the years, he produced many videotapes of his technical work. To enhance his presentations, he often brought his own materials, equipment, dental assistants (and sometimes patients), all at his own expense.

In the 1970s, Dr Markley traveled to Spain and gave multiple courses in operative dentistry throughout the country. His influence on Spanish dentistry (then and now) is nothing short of astounding! Within a short period of time, his guiding principles were being taught in dental schools and accepted by dental practitioners.

Over the years Dr Markley received many awards, including the 1975 Outstanding Achievement Award from the American Society for Preventive Dentistry and the 1978 Annual Hollenback Prize by the Academy of Operative Dentistry. On August 16, 1983, in Washington, DC, he received the Department of Defense Medal for Distinguished Public Service for his outstanding efforts in training military dentists. The Chiefs of the Tri-Services were all present. In 1988, a nine-chair clinic at the University of Colorado Dental School was dedicated in his name and a scholarship fund was established

Dr Markley was preceded in death by his wife of 59 years, Winnifred. He is survived by a son, John L Markley of Madison, Wisconsin; a daughter, Marian R Markley of Ridgefield, Washington; a brother, Robert E Markley of McMinnville, Oregon; and three grandchildren. On Saturday, March 4, 2000, a memorial service was held at the Park Hill United Methodist Church, Denver. Memorial contributions may be made to the Miles R Markley Scholarship Fund, University of Colorado Health Sciences Center, c/o CU School of Dentistry, Campus Box 284-19, 4200 E Ninth Ave, Denver, CO 80262.

Arden G Christen

Information for the above "In Memoriam" was taken, in part, from Dr Christen's *Aeromedical Review* 1-78 entitled, "Life Story of a Prevention-Oriented Dentist: An Interview with Miles R Markley, DDS" (October 1978). Dr Christen is making available a limited number of complimentary copies of this 65-page publication that will be sent to interested individuals on a first-come, first-served basis. Dr Christen may be reached at the following address:

Arden G Christen, DDS, MSD, MA
Indiana University School of Dentistry
1121 West Michigan Street
Indianapolis, Indiana 46202-5186
Internet: achriste@iupui.edu

©Operative Dentistry, 2000, **25**, 147-148

Guest Editorial

Operative Dentistry in the Academic World

When this academy was formed 29 years ago, we were excited about its potential to influence the teaching of operative dentistry, which was then steadily declining as a strong clinical discipline. Many of the charter members of this academy held the general belief that our field of operative dentistry should be recognized as a specialty in its own right. The founding of the American Board of Operative Dentistry was viewed as a giant step forward in the movement to return operative dentistry to its rightful place as a primary clinical discipline.

Our academy has grown and been very active and we can take pride in its accomplishments. The questions we must ask ourselves are: "Have we done enough?" "Has the quality of teaching in operative dentistry increased across this country?" "What has been happening over the past 29 years within our dental schools?" "Are the deans of these institutions aware of the need to increase the time allocated for this discipline?" And, "Are there an adequate number of fully-qualified operative dentists on the faculty?" To further complicate these issues, all schools are having difficulty funding faculty positions, and the salaries for such faculty are woefully inadequate.

Operative dentistry continues to vary from school to school. This is noted by the pass/fail ratio of graduating students from one school to another as their graduates challenge the various regional boards. Some schools have a very high pass rate—in some years no students fail a regional board exam. Others, however, have a dismal record when challenging the same examination. Doesn't that tell us something?

How many faculty teaching operative dentistry are doing so because they are recognized experts in this discipline or because the administration thinks that any general dentist is adequate to be a faculty member for operative dentistry. Don't take me wrong, many general dentists really are the best teachers we have.

We have seen much of the operative curriculum taken over by our fixed prosthodontists. At one time all single unit restorations were part of the operative curriculum, including inlays, onlays, and crowns. The specialty status achieved for prosthodontics has led to their dominating the teaching of all indirect restorations in most school curriculums. As recognized specialists, they were able to prevent the teaching of any inlays within their schools because they erroneously believed that inlays split teeth. When schools removed all indirect restorations from operative dentistry, it spelled the death knell for operative dentistry.

With diminished curriculum time and lack of specialty recognition, direct and indirect intracoronal restorations are relegated to an inferior position for which there is little respect. In this country most students today, and that includes those here at the University of Washington, feel that full-coverage restorations are almost always the preferred treatment for teeth that require cuspal protection and think that the full crown is the only treatment available. Many feel any base is adequate for a foundation on which to place a crown. Without good operative procedures to provide the desired resistance and retention form, crown failures increase exponentially.

Schools that have reduced the number of full-time faculty teaching in our restorative clinics are ensuring a continued decline in all technical procedures. Unfortunately, there appears to be no relief in sight.

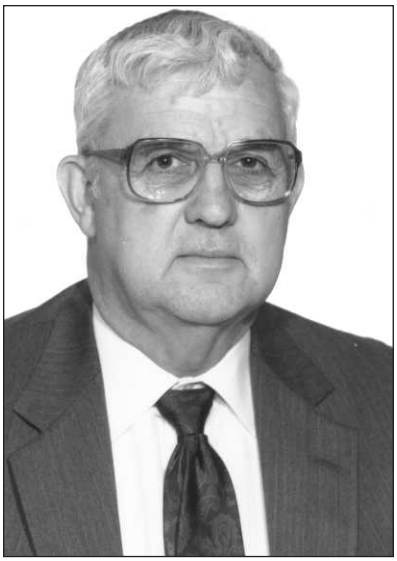
There are some bright lights about, however. Here is one example: Until last year, we had a very difficult time finding faculty who could provide our students good supervision preparing and delivering cast gold inlays/onlays. A newly-developed program here at the U of W came about when Dr Richard (Dick) Tucker, of cast gold inlay/onlay fame, volunteered three of his local study clubs to staff a special inlay clinic once each week. His study club members work one-on-one with students from the start of the preparation to the finish of the restoration. One study club group handles each of the three quarters.

I would suggest that our Academy Study Club Committee look into the feasibility of encouraging other groups to follow the model of the Tucker Inlay Clubs,

which are so effective for us in Seattle. I am not just referring to inlay clubs, but any of the other study groups dealing with direct and indirect single unit restorations.

Historically, our chosen discipline of operative dentistry has had many great leaders and teachers, some in academia, and many others who directed their colleagues in study clubs. With the recent passing of Dr Miles Markley, our profession and, in particular, members of this academy, lost one of its greatest members. He taught and advocated a very conservative cavity preparation and gave us the first successful pin system for adding retention and resistance form for badly broken-down teeth. These are but a couple of his contributions. His impact on the profession was immeasurable. Perhaps his greatest gift of all was his desire to promote excellence in cavity preparation. He was a living example of what we need to do to provide our schools with effective direction and skills. He will be missed by us all!

David J Bales
University of Washington
School of Dentistry



David J Bales

Commentary

When Dr David J Bales was hired as chairman of the Department of Restorative Dentistry at the University of Washington, it is doubtful that he anticipated the additional workload that would soon be his. Dr Ian Hamilton felt that it was time for new hands on the reins of the journal he had nurtured for 12 years and, in 1986, Dr Bales became the second editor of *Operative Dentistry*. His first editorial was a special tribute and thank you to Dr Hamilton. Although, as he learned how much commitment of time and energy his new position required, "thank you" may not have been a phrase that was foremost in his mind.

There could not have been a better choice for a new editor, however. Dave's exceptional work ethic, depth of knowledge, and dedication to his profession provided all the necessary ingredients to continue Dr Hamilton's work. For the next seven years Dr Bales guided *Operative Dentistry* to a position as a premier journal for the restorative dentist. He introduced computer technology to the journal production and, in 1990, moved the publication from four to six issues per year. During his tenure he produced seven volumes, 34 issues and two supplements, one of which was the prestigious "Proceedings of the International Symposium on Adhesives in Dentistry," which contained the 22 state-of-the-science papers presented in Nebraska. David published numerous thought-provoking editorials and also gave us a new cover for his final six issues, basically reversing the existing color scheme to a white background with blue lettering and logos.

Although he accomplished many things for the journal, Dave's greatest contribution to *Operative Dentistry* may have been the addition of his wife, Darlyne, as editorial assistant in 1987 and Kate Flynn Connolly as editorial associate in 1988. These ladies provided tremendous support, expertise, and enthusiasm to the daily business of publishing a quality journal and continued their duties until the recent move of the journal to Indiana.

Dr Bales appreciation of his task was apparent in his first Editorial (*Operative Dentistry*, 11(1), 1-2, 1986), when he included the following quote from a letter from HL Mencken to William Saroyan: "I note what you say about your aspiration to edit a magazine. I am sending you by this mail a six-chambered revolver. Load it and fire every one into your head. You will thank me after you get to Hell and learn from other editors how dreadful their job was on earth." Thankfully, Dave did not take this advice to heart and continued to produce a journal that made Dr Hamilton, the academies, and our readership very proud.

Michael A Cochran
Editor

©Operative Dentistry, 2000, **25,** 149-151

Clinical Research

The Efficacy of Reservoirs in Bleaching Trays

DS Javaheri • JN Janis

Clinical Relevance

The use of blockout spacers to create bleaching-solution reservoirs did not increase the success of home bleaching in this study.

SUMMARY

Thirty patients were selected to bleach their maxillary teeth. Vacuum-formed trays were fabricated for the maxillary arch so that only one quadrant of bleaching tray had reservoirs. Nupro Gold 10% carbamide peroxide gel was given to patients. They were instructed to place a drop of gel in each tooth area and wear the trays twice a day for two-hour periods. After 10 days the patients were evaluated for shade changes in each quadrant. No clinical difference was observed in the after-shade match of the two quadrants. It appears that the addition of blockout spacers to create reservoirs does not increase the success of home bleaching.

INTRODUCTION

Different variations of the at-home bleaching technique have been published (Haywood & Heymann, 1989; Goldstein, 1989; Darnell & Moore, 1990; Haywood, 1991a,b). Most authors and bleaching gel manufacturers recommend the use of reservoirs to increase the amount of product available for bleaching and to allow for complete seating of the bleaching tray (Dentsply-Ash, 1996; Discus Dental, 1996; Ultradent Products, 1996). The reservoirs are formed by the addition of light-cured composite spacers to the patient models prior to tray fabrication. However, more bleaching gel may not give better results, since the chemical activity of the gel may be limited by surface area and time of exposure, not by volume. The use of foam spacers has not been shown in clinical trials to shorten the treatment time or improve the efficacy of the bleaching treatment (Haywood, Leonard & Nelson, 1993). The reservoirs decrease tray retention and increase lab fabrication time and cost.

The purpose of this study was to determine if the use of reservoirs in bleaching trays would cause a difference in the final shade of the bleached teeth.

METHODS AND MATERIALS

Thirty patients, 22 women and 8 men, ranging in age from 19 to 64, were selected to bleach their maxillary teeth. The selected patients had dark yellow or yellow-brown teeth with minimal restorations. Patients with cervical abrasion/erosion, receded gums, and gray or tetracycline-stained teeth were not included in this study. The patients were informed that significant whitening of teeth can be achieved in the majority of cases, but that results could not be guaranteed. For this study, only the maxillary was whitened. When the

AEGD Residency Program, University of the Pacific Dental School, 2215 Webster St, San Francisco, CA 94115

Dino S Javaheri, DMD assistant professor

Joel N Janis, DDS, associate professor, Department of Operative Dentistry

patients presented for their 10-day bleaching evaluation, they were given extra gel and trays for the mandibular arch.

An alginate impression was made of both arches. The disinfected, rinsed impressions were poured with dental stone. The resultant casts were inspected for voids and chips. The casts were trimmed on a model trimmer to achieve a base parallel to the occlusal plane of the posterior teeth and to eliminate the land area beyond the depth of the vestibule. The base of the casts was flat with a hole in the center. The casts were allowed to dry for 24 hours prior to tray fabrication. Spacers were placed on the left quadrant of the maxillary casts (Figure 1). The reservoirs were created using a highly viscous light-cured composite gel (Triad Gel, Dentsply-Ash, York, PA 17405). The spacer was placed 1 mm from the gingival margin. Occlusal and lingual surfaces, interproximal areas, and the incisal edges were not covered. The thickness of the facial reservoirs was approximately 0.5 mm. A hand-held curing light was applied to the gel surfaces for one minute. The cured surfaces were wiped with a damp alcohol gauze pad to remove the slightly tacky surface that remained on the cured gel. A heat/vacuum tray-forming machine was used to fabricate the trays. Trays were fabricated with a 0.035" thick, 5" x 5", soft tray material (Dentsply-Ash), and had a smooth and a pebble-finished side. The whitening sheets were placed in a preheated heat/vacuum tray-forming machine with the pebble finish toward the stone model. Upon softening to the point that the material sagged about one inch, the vacuum was engaged, and the heated material was lowered slowly onto the cast to avoid generating wrinkles or folds. Ample time under the vacuum was allowed for the material to adapt to the cast. The trays were allowed to cool on the cast prior to further manipulation. Finally, the trays were trimmed approximately 3 mm beyond the teeth on the tray, creating a horseshoe design. The excess tray material was trimmed with nonserrated scissors. The trays were properly trimmed to allow for unrestricted movement of the frenum attachment.

Inspection of the internal aspect of the trays for positives and viewing through the guard for tissue blanching was done (Figure 2). The edges of the trays were also inspected for rough areas that could irritate the tissues. After the fit of the bleaching trays was determined to be satisfactory, a Vita guide shade match was done. Both dentists evaluated the teeth together. A consensus of the shade of each quadrant was required.

Patients were given a 10% carbamide peroxide gel, Nupro Gold (Dentsply-Ash), and the following instructions were reviewed. After brushing and flossing, one small drop of gel was to be placed in the labial aspect of each tooth area of the tray. Excess gel oozing out of

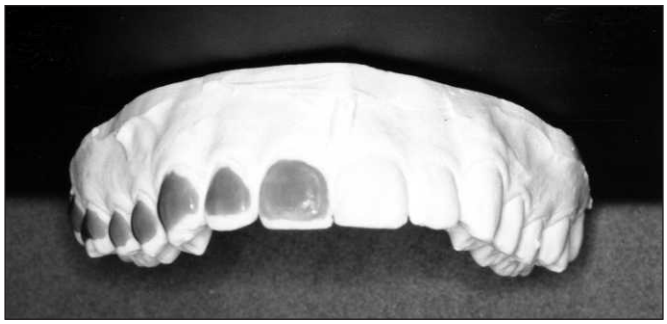


Figure 1.

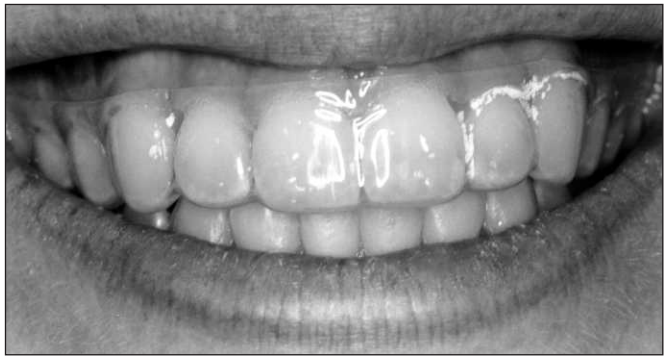


Figure 2.

the tray was removed by running a wet finger over the periphery of the tray. Patients were instructed to wear the trays twice a day for two-hour periods. Potential problems, such as gum irritation, sensitivity, and sore throat, were reviewed with the patients. Follow-up appointments were made for 10 days later. Both evaluators did Vita shade guide matching at the post-whitening appointment. Patients were questioned regarding any tooth sensitivity, gingival irritation, or other concerns.

RESULTS

No difference between the right (reservoir) quadrant and the left (non-reservoir) quadrant was observed in the post-whitening shade determination. Statistical analysis was not conducted since no difference between sides was noted on any of the patients. None of the patients reported any tooth sensitivity or mucosal irritation during the study.

DISCUSSION

In this clinical study, there was no difference between the final shade of the reservoir and non-reservoir sides. Some companies that produce home bleaching gel recommend the use of reservoirs with the theory that the extra space will allow for more bleaching solution to be available to the tooth in a continual supply. However, the true efficacy of the bleaching gel may be related to the surface area of the tooth and the time of

exposure. If the bleaching solution in the reservoirs degrades at the same rate, then more gel will not necessarily yield better results.

CONCLUSION

The presence or absence of reservoirs in a bleaching tray did not affect the results of a dentist-supervised, home-applied whitening with a 10% carbamide peroxide gel.

(Received 20 October 1998)

References

Darnell DH & Moore WC (1990) Vital tooth bleaching: The White and Brite technique *Compendium* **11(2)** 86, 88-90, 92-94.

Dentsply-Ash (1996) *Nupro Gold Total Tooth Whitening System* (instructions), Dentsply International, York, PA 17405.

Discus Dental (1996) Nite White (Instructions), Discus Dental, Inc, Los Angeles, CA 90064.

Goldstein FW (1989) New "at home" bleaching technique introduced *Cosmetic Dentistry* June 6-7.

Haywood VB (1991a) Nightguard vital bleaching, a history and products update: Part 1 *Esthetic Dentistry Update* **2(4)** 1991 63-6.

Haywood VB (1991b) Overview and status of mouthguard bleaching *Journal of Esthetic Dentistry* **3(5)** 157-161.

Haywood VB & Heymann HO (1989) Nightguard vital bleaching *Quintessence International* **20(3)** 173-6.

Haywood VB, Leonard RH Jr & Nelson CF (1993) Efficacy of foam liner in 10% carbamide peroxide bleaching technique *Quintessence International* **24(9)** 663-6.

Ultradent Products (1996) Opalescence (Instructions), Ultradent Products, Inc, South Jordan, UT 84095.

©*Operative Dentistry, 2000,* **25**, 152-154

Three-Year Clinical Evaluation of a Polyacid-Modified Resin Composite (Dyract)

MJ Tyas

Clinical Relevance

Retention of a polyacid-modified resin composite in cervical non-stress bearing areas was excellent after three years. However, marginal discoloration remains a concern.

SUMMARY

The aim of this study was to clinically evaluate a polyacid-modified resin composite (Dyract; Dentsply deTrey). Forty-one Dyract restorations were placed (36 in noncarious cervical cavities and five in anterior approximal cavities), and assessed after three years. The retention rate was 97% for the cervical restorations; however, 16 restorations showed some degree of marginal discoloration, sometimes severe. Color match and surface integrity were highly satisfactory throughout the trial. Dyract has now been superseded by Dyract AP, and the manufacturers should consider recommending mandatory enamel etching.

INTRODUCTION

Polyacid-modified resin composites were introduced in approximately 1994. They were developed in an attempt to confer some of the properties of glass-ionomer cements on resin composite materials, and are available both as single- and two-component materials. The former are commonly known as compomers, which reflects their fundamental resin composite nature, together with their claimed glass-ionomer properties.

The first compomer on the market was Dyract (Dentsply de Trey, Konstanz, Germany). Since then, other manufacturers have marketed compomers, and in early 1998, Dyract was superseded by Dyract AP (All Purpose), which is claimed to be satisfactory for all clinical situations, including posterior stress-bearing areas. The general chemistry of compomers is that part of the resin matrix is an acid resin, which ionizes by the water absorbed during the days and weeks after photopolymerization, to produce hydrogen ions. These hydrogen ions react with the filler particles, which consist of a fluoride-containing glass similar to that used in glass-ionomer cement. The result is the formation of a cross-linked polymer and the release of fluoride; however, most researchers report that the fluoride release is considerably less than that from glass-ionomer cement (Momoi & McCabe, 1993).

Adhesion to the dental hard tissues is achieved by various mechanisms, depending on the product. In the case of the original Dyract, a primer (PSA Prime) was applied to clean, unetched enamel and dentin. PSA Prime contains a phosphoric acid monomer, which is claimed to bond ionically to the calcium component of the enamel and dentin.

School of Dental Science, The University of Melbourne, Faculty of Medicine, Dentistry, and Health Sciences, 711 Elizabeth Street, Melbourne 3000 Australia

Martin J Tyas, DDSc, PhD, FRACDS, FADM

The results of a one-year study of the clinical performance of Dyract, mainly in noncarious cervical lesions, have been reported earlier (Tyas, 1998). The present report is of the three-year follow-up.

METHODS AND MATERIALS

The clinical procedure has been described previously (Tyas, 1998). Essentially, 36 nonundercut noncarious cervical lesions and five lower anterior approximal lesions were restored in nine patients of mean age 62 years (range: 50-75 years). The manufacturer's instructions (which did not include enamel etching) were followed, and color transparency photographs were taken at 1:1 magnification. Patients were recalled at one year, two years, and three years for assessment of restoration integrity and further photographs.

The color photographs were used for assessment of marginal discoloration of the cervical restorations only, on a continuous linear rating scale of 0 to 8 (Tyas, 1998). This assessment was based on a comparison of a photograph of the trial restoration with a series of standard restorations of increasing amounts of marginal discoloration. Student's *t*-tests were applied to determine whether there was a significant difference between mean marginal discoloration scores at successive time intervals.

RESULTS

All patients returned for one-year, two-year, and three-year recalls. At one year, one cervical restoration was missing; the retention rate was therefore 97%. At two years, all remaining cervical restorations were present. At three years, one further cervical restoration was lost, giving a retention rate of >95%.

The mean marginal discoloration scores for the cervical restorations at base line, one year, two years, and three years were 0.02, 0.56, 1.60, and 1.97, respectively. The increase in mean marginal discoloration from base line to one year, and from one year to two years, was statistically significant ($p<0.05$); that from two years to three years was not significant ($p=0.48$). For individual cervical restorations, the majority (18) scored zero, and there were four scores of 2, one score of 3, five scores of 4, and six scores >5. (A score of 3-4 may be considered to be of aesthetic concern).

At three years, all five approximal restorations were present and none showed any marginal discoloration.

DISCUSSION

There have been few published clinical studies on the performance of polyacid-modified resin composite (compomer) materials. Several abstracts, cited earlier (Tyas, 1998), also reported one-year retention rates of >97% in nonundercut cervical lesions. Two-year retention rates have been found to be 100% in two studies by the same first author (Abdalla & Alhadainy, 1997; Abdalla, Alhadainy & García-Godoy, 1997). The present results for retention are therefore consistent with other authors.

The bonding mechanism of PSA Prime is claimed to be ionic to the hard tissue calcium. Bond strengths using PSA Prime have been cited at between 4.2 MPa and 14.1 MPa for unetched enamel (Abate & others, 1997; Cortes, García-Godoy & Boj, 1993; Desai & Tyas, 1996; Fritz, Finger & Uno, 1996), and between 14.3 MPa and 26.1 MPa for etched enamel (Abate & others, 1997; Cortes & others, 1993; Desai & Tyas, 1996). For dentin, bond strengths of 8.3 MPa to 21.1 MPa have been published (Abate & others, 1997; Abdalla & García-Godoy, 1997; Buchalla, Attin & Hellwig, 1996; Fritz & others, 1996; García-Godoy, Rodríguez & Barbería, 1996; Peutzfeldt, 1996; Triana & others, 1994).

Although laboratory bond strengths are not necessarily predictive of clinical performance (Finger, 1988), the values cited above for dentin are clearly adequate to retain restorations in nonundercut cervical cavities, where the majority tissue is dentin. The adhesive component of PSA Prime is dipentaerythritol pentacrylate phosphoric acid monomer (PENTA), and the phosphate group is designed to bond ionically to dentin and enamel calcium. This approach was used in the early dentin bonding agents (eg, Scotchbond; 3M Dental Products, St Paul, MN 55144), but the retentive ability was very poor (Tyas & others, 1986). However, PSA Prime has an acetone solvent, which may improve the wetting of the dentin surface, and thus, the adhesive potential (Abate & others, 1997). Other factors for the high-bond strengths using Dyract/PSA Prime have also been suggested, including the possibility of the carboxyl groups in the resin matrix forming an ionic bond to calcium (similar to the glass-ionomer cement bonding mechanism), and the elastomeric resin in PSA Prime acting as a stress buffer (Abate & others, 1997). However, there does not appear to be any experimental support for these suggestions.

Most of the marginal discoloration was found at the incisal/occlusal (enamel) margin of the restoration, although this may be because the gingival margin is frequently hidden in the gingival sulcus. Nevertheless, the amount of enamel marginal staining is of concern. Despite Dyract being termed a compomer, it is basically a resin composite, as identified by the correct name for compomers, "polyacid-modified resin composites." It is well established that the bonding of resin composite to enamel is markedly improved by acid etching the enamel in order to create retentive microporosites. As discussed earlier, the manufacturers of Dyract do not recommend enamel etching on the basis that the bond strength should be adequate with the use of PSA Prime alone. The current material, Dyract AP, is now being marketed with a new primer. Prime&Bond NT, and acid etching of the enamel is only recommended in

cases without mechanical retention (but where the cavity walls are mainly enamel), in occlusal stress-bearing areas, and where beveled margins are preferred. The manufacturers should consider mandatory enamel etching.

Marginal discoloration in one study was observed around one of 28 restorations of Dyract after two years (Abdalla & others, 1997), and in another study there was no marginal discoloration among 18 restorations after two years (Abdalla & Alhadainy, 1997). However, United States Public Health Service Bravo scores for marginal adaptation were recorded for 6% and 16% of restorations, respectively.

The results of the present study are not necessarily predictive of the clinical performance of Dyract AP. According to the manufacturers, several changes have been made to Dyract to produce the newly-introduced Dyract AP, including a finer filler particle, the addition of a cross-linking resin, and an optimized initiator system. This system needs to be clinically evaluated to determine the best procedure for ensuring maximum marginal adapation of Dyract AP.

CONCLUSIONS

Dyract exhibited excellent retention in nonundercut cervical cavities. However, there were concerns regarding the frequency and extent of enamel marginal staining. The manufacturers should consider specifying mandatory enamel etching.

(Received 5 November 1998)

References

Abate PF, Bertacchini M, Polak MA & Macchi RL (1997) Adhesion of a compomer to dental structures *Quintessence International* **28(8)** 509-512.

Abdalla AI & Alhadainy HA (1997) Clinical evaluation of hybrid ionomer restoratives in Class V abrasion lesions: Two-year results *Quintessence International* **28(4)** 255-258.

Abdalla AI, Alhadainy HA & García-Godoy F (1997) Clinical evaluation of glass ionomers and compomers in Class V carious lesions *American Journal of Dentistry* **10(1)** 18-20.

Abdalla I & García-Godoy F (1997) Bond strengths of resin-modified glass ionomers and polyacid-modified resin composites to dentin *American Journal of Dentistry* **10(6)** 291-294.

Buchalla W, Attin T & Hellwig E (1996) Influence of dentin conditioning on bond strength of light-cured ionomer restorative materials and polyacid-modified composite resins *Journal of Clinical Dentistry* **7(4)** 81-84.

Clinical Research Associates (1996) Dentin-resin adhesion, newest systems *CRA Newsletter* **20(5)** 1-3.

Clinical Research Associates (1996) Dentin-resin adhesion, 5 newest systems *CRA Newsletter* **21(6)** 1-2.

Cortes O, García-Godoy F & Boj JR (1993) Bond strength of resin-reinforced glass ionomer cements after enamel etching *American Journal of Dentistry* **6(6)** 299-301.

Desai M & Tyas MJ (1996) Adhesion to enamel of light-cured poly-acid dental materials *Australian Dental Journal* **41(6)** 393-397.

Douglas WH (1989) Clinical status of dentine bonding agents *Journal of Dentistry* **17(5)** 209-215.

Eick JD, Gwinnett AJ, Pashley DH & Robinson SJ (1997) Current concepts on adhesion to dentin *Critical Reviews in Oral Biology and Medicine* **8(3)** 306-335.

el Kalla IH & García-Godoy F (1998) Bond strength and interfacial micromorphology of four adhesive systems in primary and permanent molars *Journal of Dentistry for Children* **65(3)** 169-176.

Finger WJ (1988) Dentin bonding agents. Relevance of *in vitro* investigations *American Journal of Dentistry* **1** Spec No:184-188.

Fritz UB, Finger WJ & Uno S (1996) Resin-modified glass ionomer cements: bonding to enamel and dentin *Dental Materials* **12(3)** 161-166.

García-Godoy F, Rodriguez M & Barberia E (1996) Dentin bond strength of fluoride-releasing materials *American Journal of Dentistry* **9(2)** 80-82.

May KN Jr, Swift EJ Jr & Bayne SC (1997) Bond strengths of a new dentin adhesive system *American Journal of Dentistry* **10(4)** 195-198.

May R (1998) Dentin tensile bond strength: effect of different primer application *Journal of Dental Research* **77** 638 Abst No 50.

Momoi Y & McCabe JF (1993) Fluoride release from light-activated glass ionomer restorative cements *Dental Materials* **9(3)** 151-154.

Nakabayashi N, Nakamura M & Yasuda N (1991) Hybrid layer as a dentin-bonding mechanism *Journal of Esthetic Dentistry* **3(4)** 133-138.

Ølio G & Olsson S (1990) Tensile bond strength of dentin adhesives: a comparison of materials and methods *Dental Materials* **6(2)** 138-144.

Peutzfeldt A (1996) Compomers and glass ionomers: Bond strength to dentin and mechanical properties *American Journal of Dentistry* **9(6)** 259-263.

Triana R, Prado C, Garro J & García-Godoy F (1994) Dentin bond strength of fluoride-releasing materials *American Journal of Dentistry* **7(5)** 252-254.

Tyas MJ (1998) Clinical evaluation of a polyacid-modified resin composite (compomer) *Operative Dentistry* **23(2)** 77-80.

Tyas MJ, Burns GA, Byrne PF, Cunningham PJ, Dobson BC & Widdop FT (1986) Clinical evaluation of Scotchbond: one-year results *Australian Dental Journal* **31(3)** 159-164.

Tyas MJ (1993) Clinical testing—has it proved the efficacy of adhesives? *International Dental Journal* **43(4)** 343-347.

van Dijken JWV (1997) Clinical effectiveness of nine dentin adhesives in Class V cervical lesions *Journal of Dental Research* **76** 185 Abst No 1369.

©Operative Dentistry, 2000, **25,** 155-162

Short-Term Dentin Bridging of Mechanically-Exposed Pulps Capped with Adhesive Resin Systems

Y Kitasako • S Shibata
PNR Pereira • J Tagami

Clinical Relevance

Adhesive resin systems can protect mechanically-exposed pulps, allowing dentin bridging to occur.

SUMMARY

Dentin bridging of 150 mechanically exposed monkey pulps to two adhesive resins [BondWell LC (BW); Clearfil Liner Bond II (LB)] and a calcium hydroxide cement [Dycal (DY)] were histopathologically evaluated at 3, 7, 14, 30, and 60 days after operation (n=10). The dentin bridge structure was three-dimensionally reconstructed from serial sections using a computer-aided reconstruction system. At three and seven days, in all pulps, no necrotic tissue and slight inflammatory cell infiltration was observed just below the exposure site. At 14 days, spindle-shaped fibroblast cells could be detected at the wound surface. All dentin chips showed reparative dentin deposition along the periphery of the wound surface. From this stage, the formation of secondary dentin from the pulpal wall at the periphery of the exposed area was recognized in all pulps. At 30 days, initial signs of dentin bridging were observed at the wound surface with a well-organized layer of odontoblastoid cells. The exposed area became occluded with a dentin bridge as the observation period increased. Group DY showed significantly higher incidence of dentin bridging than other groups at 30 days ($p<0.05$). However, no significant difference of dentin bridge formation was found between Group DY and Groups BW and LB at 60 days. Bacterial penetration along the cavity walls and pulp tissue could not be detected in all groups. Histopathological observations and three-dimensional image analysis suggested that dentin bridge formation may occur following three patterns: (1) formed from the periphery of the residual dentin chip at the wound surface within 14 days, (2) formed within 14 days from the periphery of the cavity floor and with formation of reparative dentin by stimulation during the cavity preparation, and (3) formed from the wound surface within 30 days after exposure.

Department of Operative Dentistry, Faculty of Dentistry, Tokyo Medical and Dental University, 5-45 Yushima 1-chome, Bunkyo-ku, Tokyo 113-8549, Japan

Yuichi Kitasako, DDS, PhD, research associate

Shunichi Shibata, DDS, PhD, associate professor, Department of Oral Anatomy

Patricia NR Pereira, DDS, PhD, research associate

Junji Tagami, DDS, PhD, professor and chairman

INTRODUCTION

Mechanical pulp exposure results in the destruction of the odontoblasts, cell-free and cell-rich zones, as well as of varying amounts of subjacent pulpal tissue at the exposure site. Although the original odontoblasts at the exposure site are destroyed, dentinogenesis can be sec-

ondarily initiated during pulp repair (Baume, 1980). Differentiation of dental pulp cells into elongated, polarized cells forming predentin can also occur in the absence of a basement membrane and dental epithelium (Tziafas & others, 1992). Some autoradiographic studies have indicated that odontoblast replacement occurred from underlying pulp cells in the process of differentiation (Fitzgerald, 1979; Yamamura, 1985; Fitzgerald, Chiego & Heys, 1990). Yamamura (1985) indicated that injury caused by partial pulpotomy leads to either dedifferention of mesenchymal cells in the deep pulp into undifferentiated mesenchymal cells, which subsequently redifferentiate into new odontoblasts, or to direct induction and differentiation of existing undifferentiated mesenchymal cells. These studies have also described the healing of the exposed pulp as being composed of many overlapping stages, beginning with an initial inflammatory stage and ending with differentiation of cells into functioning odontoblasts and subsequent dentin bridge formation.

The mechanically exposed dental pulp possesses an inherent healing capacity for cell reorganization and dentinal bridge formation when a bacterial seal is provided. Studies of germ-free and conventional rats indicate that the presence of bacteria is a significant factor in prohibiting healing of pulp exposures (Kakehashi, Stanley & Fitzgerald, 1965). Cox and others (1987) reported that restorative materials previously reported as "toxic" did not cause pulp inflammation or necrosis when placed directly on exposed pulps as long as bacterial infection was eliminated.

There are many commercially available dental adhesive resin systems with different components and adhesive properties that may affect the healing of an exposed dental pulp. Several studies have been performed to evaluate the ability of adhesive resin systems that were used as direct pulp-capping materials in both clinical cases and controlled histopathological studies (Kanca, 1993; Kitasako & others, 1998). However, the short-term healing sequence, especially dentin bridging of mechanically exposed pulps, is not well understood. Moreover, some studies cautioned that histologic observation of only one section through a dentin bridge following capping of an exposed pulp was not by itself proper criteria for pulpal healing (Langeland & others, 1971; Mjör, 1972), and that there was a lack of appreciation of a three-dimensional perspective in interpreting microscopic sections. The aim of the present investigation was to assess the dentin bridging of mechanically exposed monkey pulps beneath adhesive resins and to observe the dentin bridge structure using three-dimensional imaging.

METHODS AND MATERIALS

Monkeys (*Macaca fuscata*), aged 7-8 years, were housed in facilities approved by the Tokyo Medical and Dental University. The animal-use protocol was reviewed and approved by the Screening Committee for Animal Research of the Tokyo Medical and Dental University.

Materials

Recently, adhesive resin systems have been developed to improve the quality of the bond-to-tooth structure while simplifying the clinical procedures. Two adhesive resin Liner Bond II (Kuraray Co, Osaka, Japan) and Bondwell LC (GC Co, Tokyo, Japan)–were investigated (Table 1). A hard-setting calcium hydroxide cement–Dycal (LD Caulk, Milford, DE 19963)–was used as the control.

Preparing the Specimens

Five monkeys were subjected to general anesthesia by intramuscular injection of 20 mg/kg ketamine (Ketaral; Sankyo Co, Tokyo, Japan) and intravenous injection of 10 mg/kg pentobarbital sodium (Nembutal Sodium Solution; Abbott Laboratories, Abbott Park, IL 60064). Class V cavities were prepared on the facial surfaces of 150 intact teeth using a high-speed tapered diamond bur (ISO #170; GC Corp) under water-spray coolant. The pulps were intentionally exposed with a round carbide bur (ISO #1; Shofu Inc, Kyoto, Japan) of 0.8 mm diameter at the cavity floor. Infiltration anesthesia was performed with lidocaine hydrochloride containing 1:80,000 epinephrine (Xylocaine; Fujisawa Co, Osaka, Japan) to control hemorrhage and exudation from exposed pulp. After alternating irrigation with 3% hydrogen peroxide and 6% solution of sodium hypochlorite three times to remove cutting debris, all exposed pulps from the same quadrant including both single and multi-roots were capped with each of the three capping materials as follows.

Bond Well LC (Group BW)

The cavity walls and exposed pulp were treated with the conditioner for 30 seconds, rinsed with water for 10 seconds, and air-dried for 15 seconds. The entire cavity and the exposed pulp were treated with the primer, air-dried, and coated with a bonding agent. The bonding agent was light cured for 20 seconds. Each cavity was restored with a hybrid resin composite listed in Table 1 and light cured for 30 seconds.

Liner Bond II (Group LB)

The cavity and exposed pulp were conditioned with a mixture of LB Primer A and B for 30 seconds, gently air-dried, and then coated with LB Bond, which was light cured for 20 seconds. A low-viscosity resin composite, Protect Liner F, was applied to the cavity walls and light cured for 20 seconds. Each cavity was restored with a hybrid resin composite listed in Table 1 and light cured for 30 seconds.

Dycal (Group DY)

Base and catalyst were mixed and directly applied on the exposed pulp. Cavity walls were left uncovered by

Table 1: *Chemical formulations of two adhesive resin systems and a hard-setting calcium hydroxide cement*

Code	Resin Bonding Systems	Materials	Compositions	Batch #	Manufacturer
BW	Bond Well LC	Conditioner	10% Citric acid, $FeCl_3$, water	200741	GC Co Tokyo, Japan
		Primer	Organic acid, HEMA, water, CQ	210741	
		Bonding agent	UDMA, HEMA, CQ	190741	
		Estio LC	Semihybrid resin composite	A3:9407143	
LB	Liner Bond II	LB Primer A	Phenyl-P, 5-NMSA, ethanol, CQ	0017	Kuraray Co Ltd Osaka, Japan
		LB Primer B	HEMA, water	023	
		LB Bond	BIS-GMA, HEMA, MDP, microfiller (15w/w %),CQ	0023	
		Protect Liner F	Low-viscosity resin composite	0007	
		Clearfil P-X	Semihybrid resin composite	A3: 0048	
DY	Dycal	Base	Substitute phenol, titanium dioxide, calcium sulfate, pigment	023410	LD Caulk Milford, DE 19963
		Catalyst	Calcium hydroxide, stearate, plasticizer, zinc oxide	023411	

BIS-GMA - bisphenol-glycidyl methacrylate; CQ = Camphorquinone; $FeCl_3$ = ferric chloride; HEMA = 2-hydroxyethl methacrylate; MDP = 10-methacryloyloxy decyl dihydrogenphosphate; Phenyl-P = 2-methacryloyloxy ethl phenyl hydrogenphosphate; UDMA = urethane dimethacrylate; 5-NMSA = N-methacryloyl-5-aminosalicylic acid.

the cement. After the cement had set, each cavity was sealed with an adhesive system (Clearfil Liner Bond II; Kuraray Co). Cavities were conditioned with LB Primer for 30 seconds, air-dried, coated with LB Bond, and light cured for 20 seconds. Protect Liner F was then applied to the cavity and light cured for 20 seconds. Each cavity was restored with a hybrid resin composite listed in Table 1 and light cured for 30 seconds.

At 3, 7, 14, 30, and 60 days after operation, the monkeys were killed by intravenous injection of 250 mg/kg thiopental sodium (Ravonal; Tanabe Pharmaceutical Co, Osaka, Japan). The teeth were extracted and the root apices removed to improve fixation. Then samples were immersed in 10% neutral buffered formalin solution for two weeks. Before immersion, the mesial and distal approximal surfaces of the teeth were reduced with a high-speed diamond stone under spray-coolant, until the dental pulp became visible through the remaining dentin, in order to facilitate the penetration of the fixing solution. The teeth were demineralized with Plank Rychlo's decalcifying solution ($AlCl_3H_2O$: 70g; 95% formic acid: 50 ml; 37% hydrochloric acid: 85 ml; distilled water: 865 ml) at 4°C for five days, then were embedded in paraffin. Five micrometer-thick histopathological serial sections of the cavities and pulp were prepared, obtaining approximately 80 to 100 sections per cavity. These were stained with hematoxylin and eosin for routine histological evaluation or with Taylor's modification of Gram's staining technique for detecting microorganisms (Taylor, 1966).

Histopathological Analysis

Histopathological changes of the exposed pulp were evaluated using a light microscope (n=10). The inflammatory cell infiltration was classified into four grades: none, slight, moderate, and severe (Mjör & Tronstad, 1972). No reaction was characterized by the absence of inflammatory cells. Slight reaction was a scattering of a small number of inflammatory cells. Moderate reaction was characterized by a distinct increase in inflammatory cell numbers. Severe reaction was characterized by a microabscess. The results of the inflammatory cell infiltration were statistically analyzed by Fisher's exact probability test (Siegel & Castellan, 1988) for differences between Dycal and the adhesive resin systems at each time interval (n=10). Dentin bridging was one of four different types (I.DB: Initial Dentin Bridging; P.DB: Partial Dentin Bridging; A.C.DB: Almost Complete Dentin Bridging; C.DB: Complete Dentin Bridging) by measuring the rate of dentin bridge formation in relation to the diameter of the exposed area. The results of dentin bridging were statistically analyzed by the Mann-Whitney U test (Siegel & Castellan, 1988) for differences between Dycal and the adhesive resin systems at each time interval (n=10).

Table 2: *Results of the Histopathological Findings*

Time Intervals Experimental Groups # of Specimens		3-Day BW 10	3-Day LB 10	3-Day DY 10	7-Day BW 10	7-Day LB 10	7-Day DY 10	14-Day BW 10	14-Day LB 10	14-Day DY 10	30-Day BW 10	30-Day LB 10	30-Day DY 10	60-Day BW 10	60-Day LB 10	60-Day DY 10
Inflammatory Cell Infiltration	none	5	5	2	8	5	5	7	4	6	8	7	7	9	9	6
	slight	5	5	8	2	5	5	3	6	4	2	3	3	1	1	4
Dentin Bridging	none				8	8	6	3	4	3	0	0	0	0	0	0
	I.DB				2	2	3	3	5	4	1	2	0	0	0	0
	P.DB						1	4	1	2	5	3	1	1	1	2
	A.C.DB									1	3	4	6	6	5	3
	C.DB										1	1	3	3	4	5

Three-dimensional Image Construction of Dentin Bridge

All single-rooted samples (n=60) were used for the three-dimensional image construction of a dentin bridge. The dentin bridge structure was three-dimensionally reconstructed from 80 sections of serial sections from each tooth using a computer-aided reconstruction system (Full Color Image Processor SPICCA-II; Avio, TRI/A-PC; Ratoc, Tokyo, Japan). Each section was scanned serially and then superimposed to obtain the three-dimensional reconstruction. A standard position for all data was set from two-dimensional images on the screen in order to permit exact superimposition and reconstruction of the dentin bridge. Each image was matched at the standard position. The outline data input could be done by tracing the image using the mouse as displayed on the CRT monitor. The network of images was displayed on the CRT monitor, and the image series and parts were displayed by different colors on the monitor. Photographs were taken directly from the display.

RESULTS

Histopathological Findings (Table 2)

Severe inflammatory reactions of the pulp (necrosis and abscess formation) were not observed. Slight inflammatory cell infiltration, mainly consisting with leukocytes, was the main reaction (Figure 1). Macrophages were only observed in Group LB at 14 days. The control Group DY showed significantly higher incidence of dentin bridging than other groups at 30 days (U_{LB}=26 ; p<0.05; U_{BW}=23 ; p<0.05). However, no significant difference of dentin bridge formation was found between Group DY and Groups BW and LB at 60 days. Bacterial penetration along the cavity walls and pulp tissue could not be detected in any group.

At three days after operation, no necrotic tissue at the superficial portion of all exposed pulps was observed (Figure 1). The disruption and absence of odontoblasts below the cut tubules were observed. Slight inflammatory cell infiltration with hemorrhage was observed just below the exposure site. A number of dentin chips were often seen in the pulp tissue.

At seven days, in two of 10 cases of Group DY, spindle-shaped fibroblast cells could be detected at the wound surface (Figure 2A). For the adhesive resins, slight inflammatory cell infiltration with hemorrhage was mainly observed just below the exposure site (Figure 2B). Differentiated cells with dark-stained nuclei were seen at the periphery of all dentin chips beneath the wound surface (Figure 2C).

At 14 days, all specimens had either no inflammation or displayed only small infiltrates close to the exposure site, and showed reparative dentin with odontoblasts along the pulpal walls where the dentin tubules had

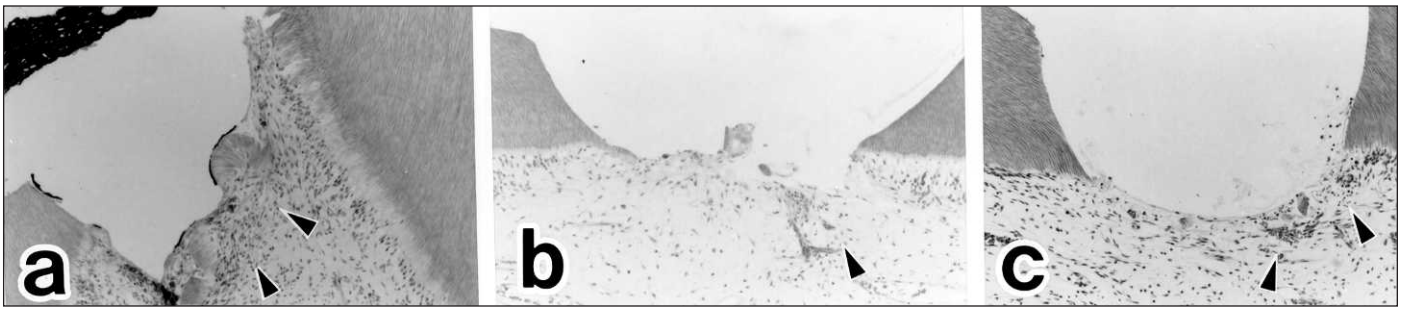


Figure 1. *Three days after operation. (a) Group DY, (b) Group LB, (c) Group BW; no necrotic tissue at the superficial portion of the exposed pulp was observed. Slight inflammatory cell infiltration (arrowheads) was observed just below the wound area. Occasional dentin chips were present at the surface of the exposed area (magnification X160).*

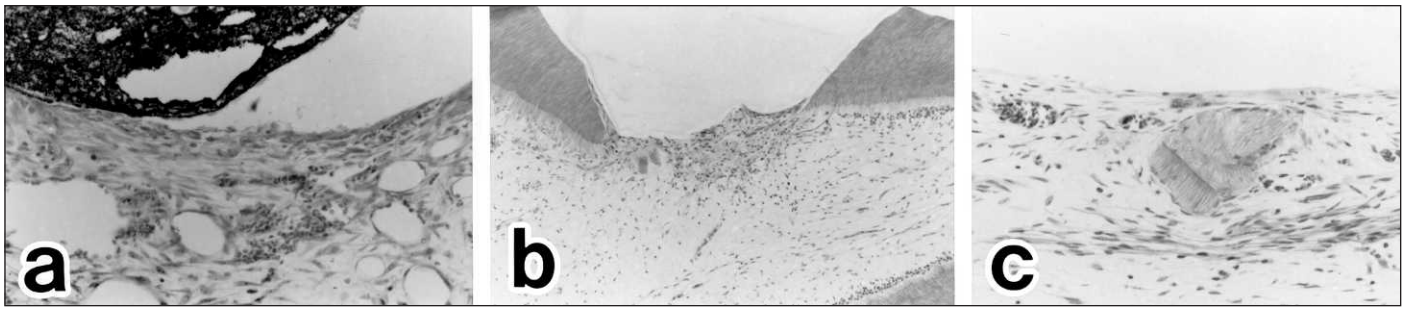


Figure 2. *Seven days after operation. (a) Group DY; spindle-shaped cells could be detected at the wound surface (magnification X320). (b) Group LB; slight inflammatory cell infiltration was mainly observed just below the exposure site (magnification X100). (c) Group BW; differentiated cells with dark stained nuclei were seen at the periphery of dentin chips (magnification X320).*

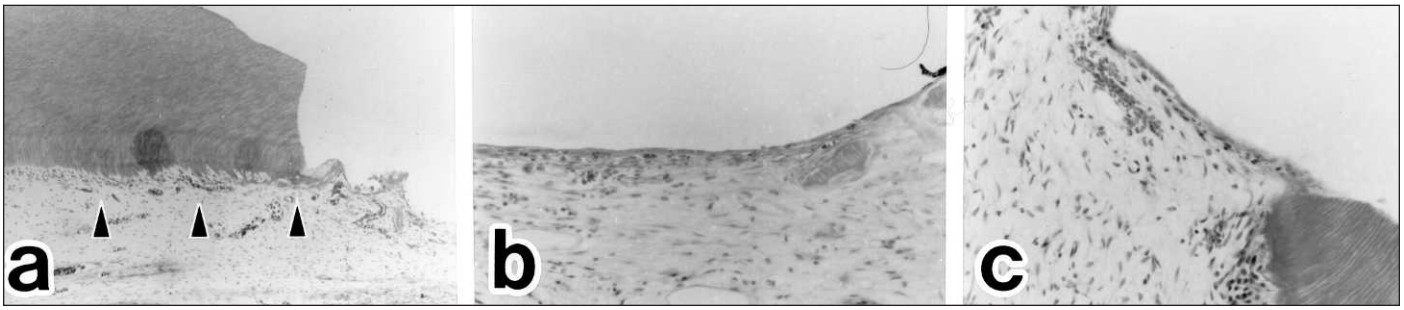


Figure 3. *Fourteen days after operation. (a) Group LB; reparative dentin (arrowheads) with odontoblasts along the pulpal walls where the dentin tubules had been cut underneath the cavity preparation was observed (magnification X100). (b) Group DY, (c) Group BW; cells elaborating in a matrix were observed as odontoblastoid cells resembling primary odontoblasts on the wound surface (magnification X320).*

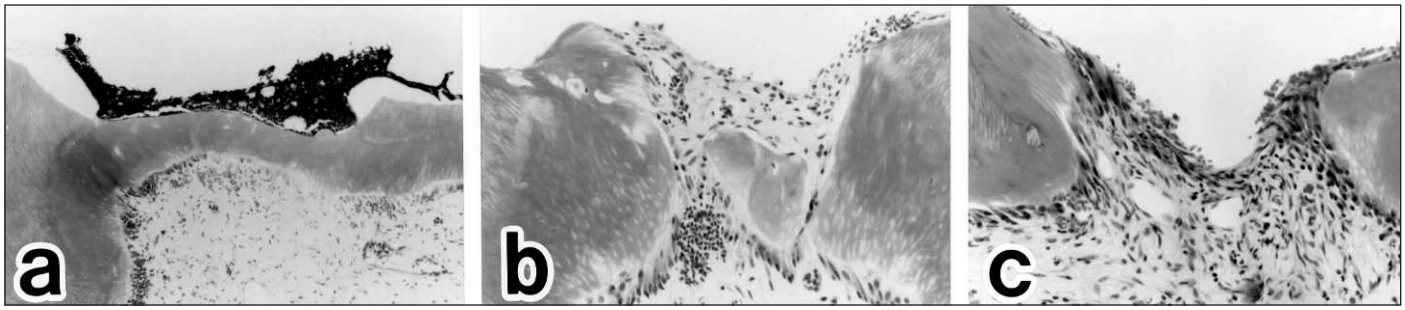


Figure 4. *Thirty days after operation. (a) Group DY; complete dentin bridging directly adjacent to the interface of the capping material was observed (magnification X160). (b) Group LB: partial dentin bridging was observed. (c) Group BW initial signs of dentin bridging were observed with a well-organized layer of cells which were similar to the odontoblasts (magnification X320).*

Figure 5. *Sixty days after operation. (a) Group DY, (b) Group LB, (c) Group BW; complete dentin bridging including a number of dentin chips were observed (magnification X160).*

been cut beneath the cavity preparation (Figure 3A). All dentin chips at the wound surface showed reparative dentin deposition along the periphery. Some dentin chips were connected to each other. Cells elaborating a matrix were often identified with odontoblastoid cells just below the exposure site (Figure 3B-C).

At 30 days, a few cases showed minimal inflammatory response, which was limited to the area beneath the exposure. The control Group DY showed dentin bridge formation directly adjacent to the interface of the medicament, and significantly higher incidence of dentin bridging than the other groups (U_{LB}=26; p<0.05; U_{BW}=23 ; p<0.05)(Figure 4A). For the adhesive resins, dentin bridge formation in varying degrees was observed (Figure 4B). Moreover, initial signs of dentin bridging were observed at the wound surface with a well-organized layer of odontoblastoid cells (Figure 4C).

At 60 days, several cases showed a minimal inflammatory response, which was limited to the area beneath the exposure. Dentin bridges, which were classified as complete dentin bridging, were observed in 20% of the specimens of all groups. No significant difference for dentin bridge formation was found between Group DY and the adhesive resin groups (p>0.05) (Figures 5A-C).

Three-dimensional Image Construction of Dentin Bridge

The exposed area became occluded with a dentin bridge as the observation period increased in all groups. Figure 6 shows three-dimensional reconstructed images of a dentin bridge of Group LB at 14 days (6A,D), 30 days (6B,E), and 60 days (6C,F) after operation. The cavity floor (CF), dentin chips (DC), and dentin bridge are apparent in Figure 6. At 14 days all dentin chips at the wound surface showed reparative dentin deposition along the periphery. Some dentin chips were connected to each other (Figure 6A). Moreover, the dentin bridge formation occurred with reparative dentin by stimulation during the cavity preparation were observed (Figures 6A and 6D). The exposed area became occluded with a dentin bridge as the observation period increased (Figures 6B and 6E). Finally, dentin bridges, which were classified as complete dentin bridging, were observed in Figure 6C and 6F. From the three-dimensional image analysis, dentin bridge formation beneath the adhesive resin mainly occurred with reparative dentin by stimulation during cavity preparation.

DISCUSSION

Studies of germ-free and conventional rats indicated that the presence of bacteria was a significant factor in inhibiting healing of pulp exposures (Kakehashi & others, 1965). The present study indicated that a slight inflammatory cell infiltration was the main reaction of

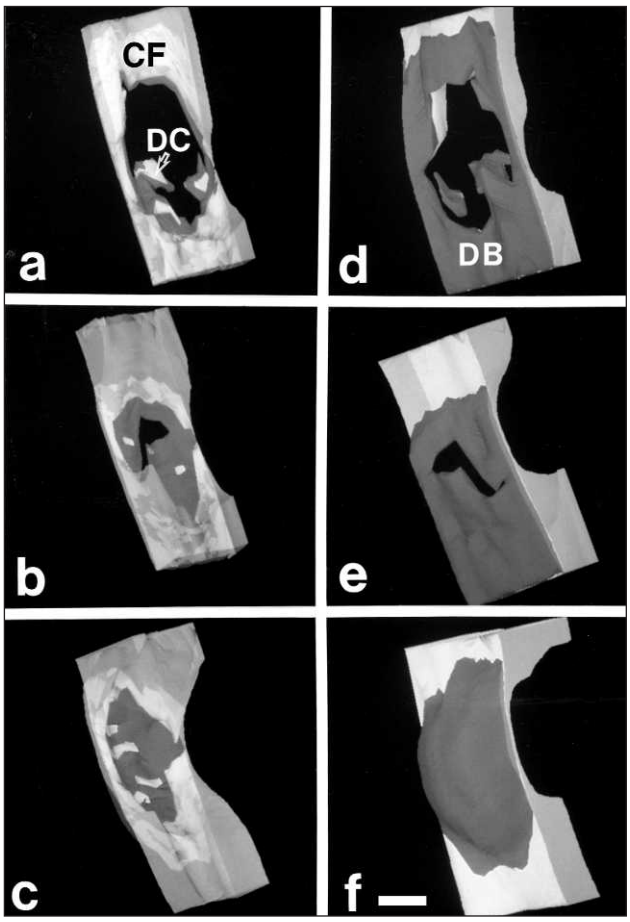


Figure 6. *Three-dimensional reconstructed image of dentin bridge of Group LB at 14 days (a,d), 30 days (b,e), and 60 days (c,f) after operation: The images (a,b,c) are those viewed from the outside of the pulp chamber through the cavity floor at an inclination of approximately 45°; and the images (d,e,f) are those viewed from the pulp side at an inclination of approximately 45°. From the three-dimensional image analysis, dentin bridge formation beneath the adhesive resin mainly occurred with reparative dentin by stimulation during cavity preparation (bar=250 μm). CF, cavity floor; DB, dentin bridge; DC, dentin chip.*

the mechanically exposed pulp when adhesive resins were used as direct pulp capping materials. The rationale for this result is based on the effective seal against bacterial invasion that was provided by adhesive resins, allowing pulpal healing to occur. The dentin bridge structure was three-dimensionally observed by applying a computer-aided reconstruction system. Three-dimensional image reconstruction of dentin bridges was effective to visualize the morphology of bridging formation.

The mechanical pulp exposure by a rotating carbide bur caused only minor structural damage in the present study. At three days, a slight inflammatory cell infiltration was the main reaction observed in all cases. In two of 10 cases of Group DY at seven days, spindle-shaped fibroblast cells could be detected at the wound surface. The same phenomenon was observed in Groups LB and BW at 14 days. At 30 days, a few cases showed a minimal

inflammatory response, which was limited to the area beneath the exposure. Moreover, initial signs of dentin bridging were observed at the wound surface with a well-organized layer of odontoblastoid cells. The wound area became occluded with a dentin bridge as the observation period increased. During reparative dentinogenesis, in the absence of a basement membrane, the adhesion of pulpal cells to an appropriate surface might be the critical requirement for the appearance of elongated, polarized, odontoblastoid cells (Veis, 1985). These odontoblastoid cells differentiated into young odontoblasts and were capable of DNA synthesis and formed a predentin-like collagen matrix (Feit, Metelova & Sindelka, 1970; Ruch, 1985; Mjör, Dahl & Cox, 1991; Tziafas & others, 1992). It is necessary to keep the initial pulpal inflammation provoked by the capping materials to a minimum, and to promote the wound surface to which pulp cells will attach and differentiate into odontoblasts at an early stage.

In the present study, dentin chips were often present in the subjacent pulp. At seven days, in all samples with dentin chips, differentiated cells with dark-stained nuclei were recognized at the periphery of the dentin chips. Dentin chips have predentin in their structure. The ability of predentin to induce odontoblastic differentiation has been well documented (Héritier, Dangleterre & Bailliez, 1989). Tziafas and others (1992) suggested that the physico-chemical properties of a surface to which pulp cells can attach is a critical requirement for expression of their odontoblastic potential during dentinal matrix-pulp tissue interactions. The residual dentin chip at the wound surface might be effective in promoting the wound surface to which pulp cells will attach and differentiate into odontoblasts.

On the other hand, it was reported that the presence of dentin chips and fragments disturbed the healing of exposed dental pulps (Kalnins & Frisbie, 1960). The complete removal of dentin chips from the operation site was difficult in the present study, even after repeated and alternate irrigation had been conducted with hydrogen peroxide and sodium hypochlorite. Since intentional exposure of healthy pulp is more favorable for wound healing than a carious exposed pulp, which might contain infected dentin chips within the exposed area, the dentin chips seemed to play a role in the dentin bridge formation in the present study.

Clarke (1970) postulated that a complete ring of dentin bridge encircles the wound in a circumferential manner from the periphery of the exposure site, and that the dentin bridge fills in towards the center of the wound. In the present study, at three days the absence of odontoblasts within the dentin tubules at the periphery of the exposed area was observed. The formation of reparative dentin from the pulpal wall at the periphery of the exposed area was not recognized in all groups at seven days. However, by 14 days, this phenomenon was observed in all groups. It was suggested that dentin bridge formation occurred with reparative dentin by stimulation during the cavity preparation, and that this phenomenon would start between 7-14 days following the mechanical exposure of monkey pulps. From the three-dimensional image analysis, dentin bridge formation occurred mainly from the periphery of the exposed site, which was associated with formation of reparative dentin by stimulation during cavity preparation. The prevention of the dentin bridge formation occurred from the periphery of the exposed site because of the presence of uncured capping agents at the periphery of the exposed site. This might generate the delay of complete dentin bridge formation.

Schröder and Granath (1971) speculated that a calcium hydroxide slurry provoked coagulation necrosis due to its high alkalinity. However, this study showed no necrotic tissue at the interface between the exposed pulp tissue and the pulp-capping material at any experimental period from 3-60 days, and odontoblastoid cells migrated in contact with the pulp capping material. Moreover, pulp healing, as well as new dentin formation, was observed directly adjacent to the material interface. This study confirmed previous observations that new hard tissue may form in direct contact with the medicament, and is not necessarily preceded by a zone of necrotic pulpal tissue (Cox & others, 1982).

Histopathological observations and three-dimensional image analysis suggested that dentin bridge formation occurred in all three patterns (Figure 7): (1) formed from the periphery of the residual dentin chip at the wound surface, (2) formed with reparative dentin by stimulation during the cavity preparation, and (3) formed from the wound surface. Moreover, the three-dimensional image analysis showed that dentin bridge formation beneath adhesive resins occurred mainly in pattern (2). However, in the three-dimensional image analysis, pattern (3) was not clearly observed. Further microscopic studies evaluating specifically the dentin bridging process from days 14-30 are required in order to investigate the pattern (3) correlation with the three-dimensional image analysis.

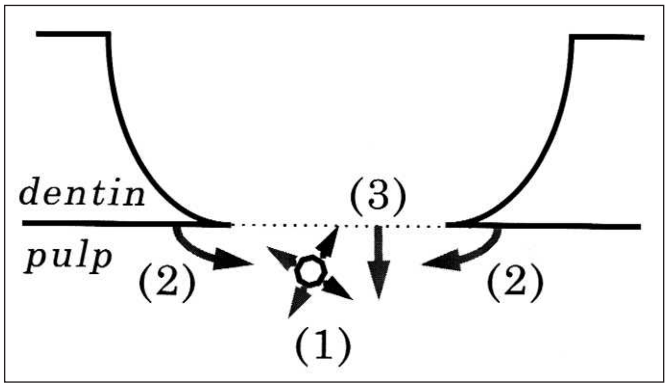


Figure 7. *Dentin bridge formation patterns; (1) formed from the periphery of the residual dentin chip at the wound surface within 14 days, (2) formed within 14 days from the periphery of the cavity floor and with formation of secondary dentin by stimulation during cavity preparation, and (3) formed beneath the capping materials by 30 days after exposure.*

CONCLUSIONS

Adhesive resin systems can protect mechanically exposed pulps, allowing dentin bridging to occur. Histopathological observations and three-dimensional image analysis suggested that dentin bridge formation may occur following three patterns: (1) formed from the periphery of the residual dentin chip at the wound surface within 14 days, (2) formed within 14 days from the periphery of the cavity floor and with formation of reparative dentin by stimulation during the cavity preparation, and (3) formed from the wound surface by 30 days after exposure.

Acknowledgments

The authors would like to thank Dr Michael F Burrow for his helpful advice in preparing the manuscript.

(Received 5 November 1998)

References

Baume LJ (1980) The biology of pulp and dentine – A historic, terminologic-taxonomic, histologic-biochemical, embryonic and clinical survey *Monographs in Oral Science* **8** 1-220.

Clarke NG (1970) The morphology of the reparative dentine bridge *Oral Surgery, Oral Medicine, Oral Pathology* **29(5)** 746-752.

Cox CF, Bergenholtz G, Fitzgerald M, Heys DR, Heys RJ, Avery JK & Baker JA (1982) Capping of the dental pulp mechanically exposed to the oral microflora–a 5 week observation of wound healing in the monkey *Journal of Oral Pathology* **11(4)** 327-339.

Cox CF, Keall CL, Keall HJ, Ostro E & Bergenholtz G (1987) Biocompatibility of surface-sealed dental materials against exposed pulps *Journal of Prosthetic Dentistry* **57(1)** 1-8.

Feit J, Metelova M & Sindelka Z (1970) Incorporation of 3H thymidine into damaged pulp of rat incisors *Journal of Dental Research* **49(4)** 783-786.

Fitzgerald M (1979) Cellular mechanics of dentinal bridge repair using 3H-thymidine *Journal of Dental Research* **58** Special Issue 2198-2206.

Fitzgerald M, Chiego DJ Jr & Heys DR (1990) Autoradiographic analysis of odontoblast replacement following pulp exposure in primate teeth *Archives Oral Biology* **35(9)** 707-715.

Héritier M, Dangleterre M & Bailliez Y (1989) Ultrastructure of a new generation of odontoblasts in grafted coronal tissues of mouse molar tooth germs *Archives Oral Biology* **34(11)** 875-883.

Kakehashi S, Stanley HR & Fitzgerald RJ (1965) The effects of surgical exposures of dental pulps in germ-free and conventional laboratory rats *Oral Surgery, Oral Medicine, Oral Pathology* **20(3)** 340-349.

Kalnins V & Frisbie HE (1960) The effect of dentine fragments on the healing of the exposed pulp *Archives Oral Biology* **2** 96-103.

Kanca J 3rd (1993) Replacement of a fractured incisor fragment over pulpal exposure: a case report *Quintessence International* **24(2)** 81-84.

Kitasako Y, Inokoshi S, Fujitani M, Otsuki M & Tagami J (1998) Short-term reaction of exposed monkey pulp beneath adhesive resins *Operative Dentistry* **23(6)** 308-317.

Langeland K, Dowden WE, Tronstad L & Langeland LK (1971) Human pulp changes of iatrogenic origin *Oral Surgery, Oral Medicine, Oral Pathology* **32(6)** 943-980.

Mjör IA (1972) Pulp reactions to calcium hydroxide-containing materials *Oral Surgery, Oral Medicine, Oral Pathology* **33(6)** 961-965.

Mjör IA, Dahl E & Cox CF (1991) Healing of pulp exposures: an ultrastructural study *Journal of Oral Pathology & Medicine* **20(10)** 496-501.

Mjör IA & Tronstad L (1972) Experimentally induced pulpitis *Oral Surgery, Oral Medicine, Oral Pathology* **34(1)** 102-108.

Ruch JV (1985) Odontoblast differentiation and the formation of the odontoblast layer *Journal of Dental Research* **64** Special Issue 489-498.

Schröder U & Granath LE (1971) Early reaction of intact human teeth to calcium hydroxide following experimental pulpotomy and its significance to the development of hard tissue barrier *Odontologisk Revy* **22(4)** 379-395.

Siegel S & Castellan NJ (1988) *Nonparametric Statistics for the Behavioral Science*, 2nd edition New York: McGraw-Hill.

Taylor RD (1966) Modification of the Brown and Brenn gram stain for the differential staining of gram-positive and gram-negative bacteria in tissue sections *American Journal of Clinical Pathology* **46(4)** 472-474.

Tziafas D, Kolokuris I, Alvanou A & Kaidoglou K (1992) Short-term dentinogenic response of dog dental pulp tissue after its induction by demineralized or native dentine, or predentine *Archives Oral Biology* **37(2)** 119-128.

Veis A (1985) The role of dental pulp–thoughts on the session on pulp repair processes *Journal of Dental Research* **64** Special Issue 552-554.

Yamamura T (1985) Differentiation of pulpal cells and inductive influences of various matrices with reference to pulpal wound healing *Journal of Dental Research* **64** Special Issue 530-540.

©Operative Dentistry, 2000, **25,** 163-169

Laboratory Research

Comparison of Conventional vs Self-Etching Adhesive Bonds to Caries-Affected Dentin

M Yoshiyama • A Urayama
T Kimochi • T Matsuo • DH Pashley

Clinical Relevance

The benefits of moist bonding can be extended to include caries-affected dentin when using Single Bond.

SUMMARY

The mechanism of resin adhesion to caries-affected dentin is still unclear. This study evaluated the interfacial morphology of two bonding systems to caries-affected dentin, coupled with the measurement of microtensile bond strengths (μTBS). Carious human molars were prepared as previously described in Nakajima and others (1995) and were bonded with Single Bond (SB) or FluoroBond (FB) according to the manufacturer's instructions, followed by creation of AP-X composite buildups. After one day of storage in 37°C water, the teeth were serially sectioned vertically into 0.8 mm slabs, trimmed to yield a 1 mm² test area, and tested to failure in a Bencor device used in an Instron machine operated at 1 mm/min. Resin-dentin interfaces were observed with SEM before or after acid/base challenge. Bonding to normal dentin with the two bonding systems (SB and FB) showed tensile bond strengths significantly higher than those to caries-affected dentin. The moist bonding technique significantly increased bond strength of SB to normal and caries-affected dentin. SEM examination revealed that typical hybrid layer and resin tags could not be formed to caries-affected dentin. The results suggested that resin penetration may be prevented by occlusion of dentinal tubules by mineral deposits that may also impart acid-resistance to the intertubular matrix of caries-affected dentin.

Department of Conservative Dentistry, School of Dentistry, Tokushima University, 3-18-15, Kuramoto-cho, Tokushima 770 Japan

Masahiro Yoshiyama, DDS, PhD, assistant professor

A Urayama, DDS, PhD, instructor

T Kimochi, DDS, postgraduate student

T Matsuo, DDS, PhD, professor and chair

David H Pashley, DMD, PhD, regents' professor, Department of Oral Biology, School of Dentistry, Medical College of Georgia, Augusta, GA 30912-1129,

INTRODUCTION

Most laboratory bonding studies are done on sound, flat, polished, freshly cut normal dentin (Gwinnett, 1993). Although such results are of great value for comparative purposes, sound, normal dentin is not the substrate most frequently encountered in clinical situations. Instead, clinicians usually must deal with caries-affected dentin or abraded-sclerotic dentin. Since these kinds of abnormal dentin frequently occur as small areas in a cavity preparation, current techniques available to test bond strength to dentin are inappropriate, as they use bonding areas that are much larger than the dimensions of most abnormal dentin. The

development of the microtensile bonding testing method (Sano & others, 1994; Pashley & others, 1995) permits measurement of tensile bond strengths in samples as small as 0.5 mm^2.

This method was recently used to measure the tensile bond strengths of several bonding systems to caries-affected dentin (Nakajima & others, 1995) and to cervical sclerotic dentin (Yoshiyama & others, 1996). In both cases, the tensile bond strengths of All-Bond 2 and Clearfil Liner Bond 2 to these abnormal forms of dentin were only about one-half as strong as the same materials to normal dentin. There was no significant difference, however, in the bond strengths of Scotchbond Multi-Purpose (SMP; 3M Dental Products, St Paul, MN 55144) to either normal or caries-affected dentin, although the values to normal dentin were lower than those obtained with the other two bonding systems. It was speculated that the 10% maleic acid used as the etchant in SMP was not acidic enough to optimally etch either type of dentin. Recently, the manufacturer has modified the bonding system into Single Bond (SB; 3M Dental Products) by mixing primer and adhesive resin components into a single-bottle product, by including 35% phosphoric acid gel as an etchant, and by recommending the use of moist bonding techniques (Kanca, 1992).

A new self-etching/self-priming adhesive system, FluoroBond (FB; Shofu, Kyoto, Japan), has been developed and marketed that produces relatively high bond strengths to normal coronal and root dentin (Yoshiyama & others, 1998). However, the adhesive properties of the new systems to caries-affected dentin have not yet been evaluated.

This study compared the interfacial morphology of the new self-etching bonding system (FB) to that of a conventional bonding system (SB) and to caries-affected vs normal dentin using scanning electron microscopy (SEM). In addition, the microtensile bond strengths (μTBS) of the systems were measured.

METHODS AND MATERIALS

Twelve extracted human molars with coronal dentin caries, stored in isotonic saline in a refrigerator, were employed in this study. The occlusal surface was ground perpendicular to the long axis of the tooth to expose a flat surface where the carious lesion was surrounded by normal dentin (Figure 1). In order to obtain caries-affected dentin, grinding was performed under running water using the combined criteria of visual examination and staining with a caries detector solution (Kuraray Co, Ltd, Osaka, Japan).

The dentin of carious lesions has often been divided into soft, nonremineralizable bacterially infected dentin and harder, remineralizable, bacteria-free affected dentin. Affected dentin is not normal because it has a lower Knoop hardness value (Nakajima & others, 1995) due to being partially demineralized. Some of that mineral precipitated in the tubules as irregular mineral deposits could interfere with resin tag formation. Infected dentin can be distinguished from affected dentin by its intense staining with caries-detector stains and by its softness. As the infected dentin is excavated by use of a round bur, the remaining affected dentin no longer takes up stain and feels firm. Such dentin commonly comprises a good deal of the pulpal floor of excavated cavities and is a clinically relevant bonding substrate.

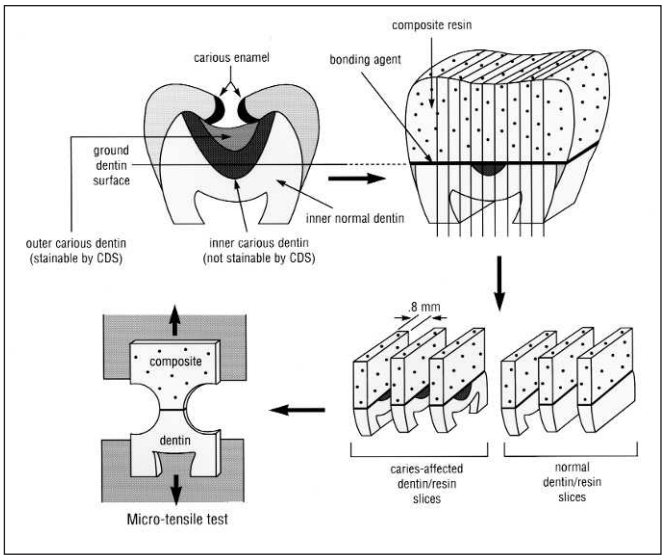


Figure 1. *Schematic illustrating the distribution of caries-affected dentin surrounded by normal dentin, the preparation of the flat surface, bonding, and the creation of a composite buildup. The buildup and underlying dentin were then vertically sectioned to create slabs. The slabs were separated into those bonded to normal vs caries-affected dentin. The slabs were trimmed to an hour-glass shape, then glued to flat stainless steel "grips" for testing tensile bond strength.*

The dentin adhesive systems used in this study are listed in Table 1. The flat prepared dentin surface was polished with #600 silicon carbide paper and then etched with 35% phosphoric acid gel for 15 seconds, and the moist specimens were bonded with SB (four teeth). After the acid-etched surface was rinsed for five seconds, excessive surface water was removed by blotting with a small piece of moist absorbent paper, leaving the surface "visibly moist" (Kanca, 1992, 1996). Additional dentin specimens (four teeth) were bonded with SB using the slightly dry bonding technique (air dried for two seconds). The four teeth used for FB bonding were not etched, slightly dried, and treated with the mixture of solutions A and B (Table 1) for 10 seconds. After air drying, adhesive was applied and light cured for 10 seconds in all specimens, and then a resin-composite crown was constructed using several layers of Clearfil AP-X composite (Kuraray Co, Ltd) to a height of 4 to 5

Table 1: *Dentin adhesive systems used in this study.*

System		Composition	Lot #
FluroBond	Solution A	H_20, photo initiator	099729
(FB Shofu	Solution B	4-AET, HEMA	099737
Kyoto, Japan)	Adhesive	4-AET, HEMA, UDMA CQ, Microfiller	099733
Single Bond	Etchant	35% phosphoric acid gel	7EC
(SB, 3M Dental	Adhesive	HEMA, Bis-GMA	7AB
Products, St. Paul		H_20, Polyalkenoic acid	
MN, 55144, USA)		copolymer	

4-AET = 4-acryloxyethyltrimellitic acid
HEMA = hydroethyl-methacrylate
UDMA = urethane dimethacrylate
CQ = camphoroquinone
Bis=GMA = bisphenol glycidyl methacrylate

Table 2: *Tensile bond strengths (MPa) of resins to normal vs caries-affected dentin.*

Material	Normal dentin	Significance	Caries-affected dentin
SB (moist	46.0 ± 10.5(9)[a]	$p<0.05$	27.1 ± 6.5(9)[b]
SB (dry)	26.4 ± 4.8(10)[b]	$p<0.05$	18.1 ± 2.1(10)[c]
FB	28.2 ± 6.1(11)[b]	$p<0.05$	17.5 ± 2.1(10)[c]

Groups identified by different superscript letters are significantly different ($p<0.05$) by Student-Newman-Keuls test. Numbers in parentheses are the numbers of specimens tested.

mm. Each layer was light cured for 20 seconds. The resin-bonded teeth were then stored in water at 37°C for 24 hours.

Depending upon the size of the tooth, five to six vertical slices approximately 0.8 mm thick were made from each tooth, parallel to the long axis of the tooth, using a low-speed diamond saw (Isomet; Buehler Ltd, Lake Bluff, IL 60044). After making the slabs, each specimen was carefully examined under a dissecting microscope to separate slabs containing resin-bonded normal dentin from slabs that contained caries-affected dentin (Figure 1). This yielded about three slabs of bonded normal dentin and two to three slabs of bonded caries-affected dentin per tooth. There were four teeth per bonding group. The slabs within a material/condition group were pooled, yielding between nine and 12 slabs per group. Under microscopic observation, the caries-affected specimens were examined to identify the regions exhibiting continuous sclerotic dentin along a 1.1 to 1.2 mm segment. The resin composite and dentin on either side of that segment were then removed with an ultrafine diamond bur in a high-speed handpiece under water spray to form an hourglass shape of the specimen (Figure 1) with the smallest dimensions at the bonded interface. The specimens of resin bonded to normal dentin were treated similarly. The specimen cross-sectional area was approximately 0.8 x 1.2 mm = 0.96 ± 0.04 mm^2, but the surface area of each failed specimen was carefully measured to calculate the bond strengths.

Three additional teeth (one per group) were used for SEM observations. That is, the carious lesions were excavated, the surface ground flat, the dentin bonded, resin composite buildups made, and five to six vertical slabs made. After trimming to an hourglass shape, the specimens were embedded in epoxy, polished with 1200-grit SiC paper, dehydrated with ascending alcohols, critical-point dried, coated with gold, and observed under the SEM. There were five to seven slabs per tooth, yielding three to four slabs of normal dentin and two to three slabs of bonded caries-affected dentin per tooth. Two or three of the normal slabs were examined without acid or base challenge. One of the polished specimens was acid etched and treated with NaOCl from each group (normal and caries-affected).

A one-way categorical ANOVA was run on the bond strengths, followed by post hoc multiple comparisons using the Student-Neuman-Keuls test and the Least Squares Means test. The latter test was used to obtain exact P values. Statistical significance was defined as $p<0.05$. All analyses were conducted with SAS software for the personal computer (SAS Institute, Cray, NC 27513).

RESULTS

The results of the microtensile bond strength tests are shown in Table 2. The bond strengths of SB to moist normal dentin were significantly higher ($p<0.05$) than bonds made to moist caries-affected dentin. Using the same material, bonds made to dry normal dentin were significantly ($p<0.05$) higher than bonds made to dry caries-affected dentin. There was no significant difference ($p=0.82$) between bond strengths made to dry normal dentin versus moist caries-affected dentin using SB. Using the self-etching bonding system (FB), the bond strengths to normal dentin were significantly higher ($p<0.05$) than those made to caries-affected dentin, using the dry bonding technique recommended by the manufacturer. There was no significant difference between bonds made with SB versus FB ($p=0.50$) on dry normal dentin or between the same two bonding systems to caries-affected dentin bonded under dry conditions ($p=0.83$). A summary of the multiple comparisons generated from the Least Squares Means test is shown in Table 3.

When moist caries-affected dentin was bonded with SB, SEM observation of the polished cross sections of the interface (Figure 2-1) showed a relatively thick

Table 3. *Multiple statistical comparison between groups.*

Groups		Least Squares Means bond Bond Strength (MPa)	Least Squares Means Probability values between groups 1	2	3	4	5	6
1.	*FBDC	17.6	-------	0.0001	0.8270	0.0015	0.0010	0.0001
2.	FBDN	28.2	0.0001	-------	0.0003	0.4984	0.6659	0.0001
3.	SBDC	18.1	0.8270	0.0003	-------	0.0029	0.0019	0.0001
4.	SBDN	26.4	0.0015	0.4984	0.0029	-------	0.8240	0.0001
5.	SBMC	27.0	0.0010	0.6659	0.0019	0.8240	-------	0.0001
6.	SBMN	46.0	0.0001	0.0001	0.0001	0.0001	0.0001	-------

*Abbreviations: FBDC = FluroBond to dry caries-affected dentin; FBDN = FluroBond to dry normal dentin; SBDC = Single Bond to dry caries-affected dentin; SBDN = Single Bond to dry normal dentin; SBMC = Single Bond to moist caries-affected dentin; SBMN = Single Bond to moist normal dentin. Multiple comparisons are done by comparing group 1 (FBDC) with groups 2-6 horizontally across the table.

hybrid layer (5-10 μm) with funneled tubule orifices. Higher magnification SEM revealed that the dentinal tubules were occupied by mineralized casts (Figure 2-2). To determine the resistance of the bonded interfaces to serial acid/base treatment, the polished caries-affected dentin specimens were treated with 10% HCl (10 seconds) followed by 5% NaOCl for two minutes. SEM of the polished cross sections of caries-affected dentin bonded with SB and etched with acid/base treatment revealed that the hybrid layer was somewhat susceptible to acid/base challenge (Figure 2-3). The top and bottom of the hybrid layer were unclear, and typical well-formed resin tags were not seen. The mineralized peritubular dentin was removed from the dentinal tubules, revealing poorly formed resin tags (Figure 2-4). In moist normal dentin, SB created typical hybrid layers and resin tags (Figure 3-1). The hybrid layers were approximately 5 μm thick, and demonstrated good resistance to acid/base challenge (Figure 3-2).

When dry caries-affected dentin was bonded with SB, SEM examination of the polished cross sections of the interface (Figure 4-1) showed a relatively thick hybrid layer. The dentinal tubules were occluded with mineralized casts (Figure 4-2). When the interfaces were etched with acid followed by treatment with sodium hypochlorite, the hybrid layers were seen to be susceptible to acid/base challenge, as was seen in moist caries-affected dentin (Figure 4-3), and small irregular resin tags were seen in the lumens (Figure 4-4). In dry normal dentin, SB created typical hybrid layers that were thinner than those in moist caries-affected dentin (Figure 5-1), but they were resistant to acid/base challenge (Figure 5-2).

When caries-affected dentin was bonded with FB, SEM examination of the polished cross sections did not reveal the presence of the hybrid layer. SEM observation of the interfaces of normal dentin bonded with FB and etched with acid and base treatment showed that the hybrid layer created by FB in normal dentin was approximately 1 μm thick and that the resin tags were

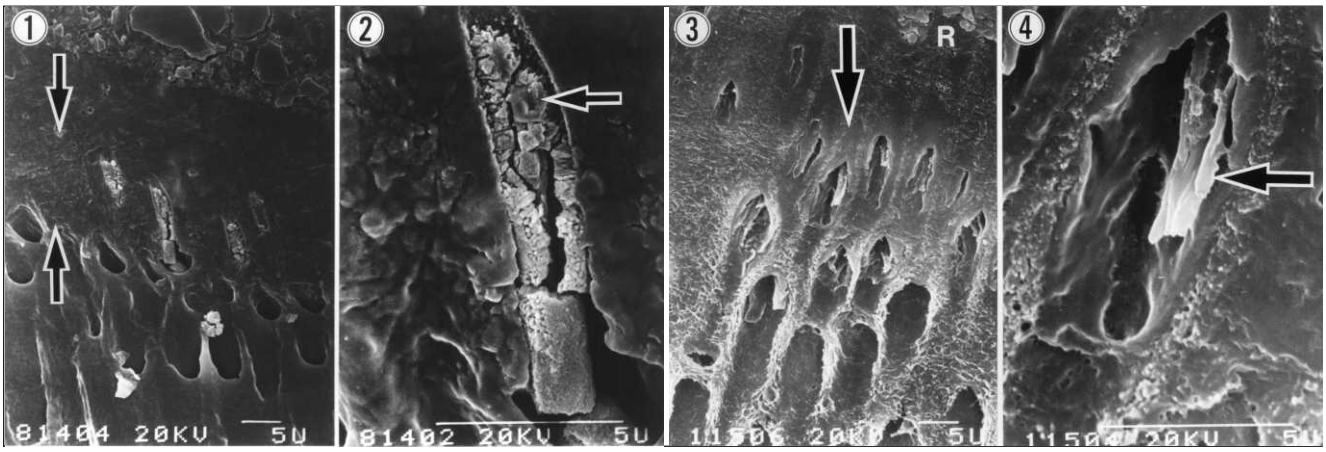


Figure 2. *SEM images of moist carries-affected dentin bonded with Single Bond (SB). (1) Moist caries-affected dentin sectioned through the bonded interface and polished. C = composite, R =resin adhesive, D = dentin. The hybrid layer (arrows) was relatively thick (5-10 µm). (2) Higher magnification of the polished interface between SB and moist caries-affected dentin. Mineralized casts (arrow) occupied the lumen of most of the dentinal tubules in the hybrid layer. (3) Moist caries-affected dentin bonded with SB, sectioned, polished, and then challenged with acid and base treatment. Note that the hybrid layer (arrow) was somewhat susceptible to acid and base challenge. (4) Higher magnification of the polished and treated interface. Mineralized peritubular dentin was removed from the tubules by the acid/base challenge to reveal poorly formed resin tags (arrow).*

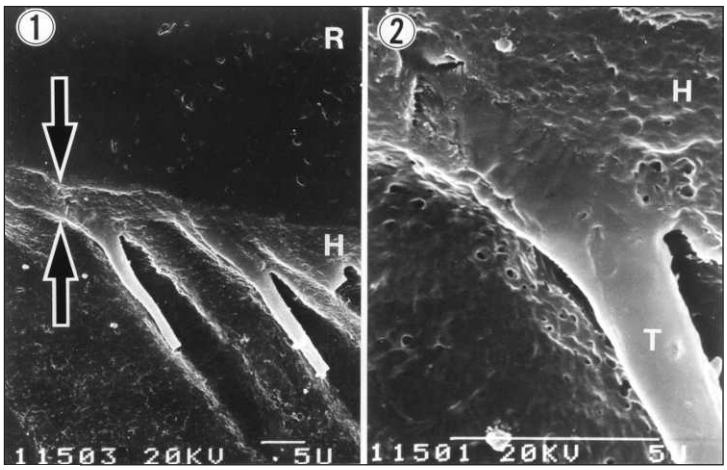


Figure 3. *SEM images of moist normal dentin bonded with SB. Abbreviations are the same as in Figure 2. (1) Moist normal dentin bonded with SB and etched with acid/base treatment. The hybrid layer (H, arrows) was about 5 µm and was resistant to acid/base challenge. (2) Higher magnification of the hybrid layer. Well-formed resin tags (T) were seen penetrating the tubules of normal dentin.*

relatively short (Figure 6-1). In addition, a transitional layer was observed below the hybrid layer with step-like structures (Figure 6-2). When caries-affected dentin bonded with FB was etched with acid followed by sodium hypochlorite treatment, there was no evidence of typical hybrid layer formation (Figure 6-3). Higher magnification SEM observations showed the absence of resin tag formation in most of the dentinal tubules (Figure 6-4). In addition, some porosities were observed in the interfaces between FB and caries-affected dentin. This self-etching adhesive was unable to penetrate into the mineral-occluded dentinal tubules of caries-affected dentin or into adjacent intertubular dentin.

DISCUSSION

The results of this work indicated that bonding to normal dentin with the two bonding systems (SB and FB) produced tensile bond strengths that were significantly higher ($p<0.05$) than those to caries-affected dentin. However, the moist bonding technique significantly increased ($p<0.05$) the tensile bond strengths of SB to both normal and caries-affected dentin.

The hybrid layers created in caries-affected dentin by the two tested bonding systems were very different, but both raise concerns. The use of 35% phosphoric acid in the SB system was sufficient to remove most of the mineral deposits from the tubules of caries-affected dentin and allowed the formation of resin tags. Because caries-affected dentin has been subjected to repeated cycles of demineralization and remineralization, it is less hard than normal dentin (Nakajima & others, 1995). Presumably, this lower hardness reflects a lower mineral content in intertubular dentin, which is already partially demineralized, making it more porous and hence susceptible to the effects of 35% phosphoric acid. This apparently results in the creation of a deeper layer of demineralized dentin after acid conditioning with 35% phosphoric acid. The adhesive resins seemed to permeate the acid-conditioned dentin to form a hybrid layer but, when cured, the layer was more susceptible to acid and base challenge than hybrid layers made with the same methods in normal dentin. This was also associated with lower tensile bond strengths. The reasons for the poorer quality hybrid layer and lower bond strengths are unclear. Perhaps more extensive water rinsing is required to solubilize the reaction products of acid conditioning of caries-affected dentin. There may be

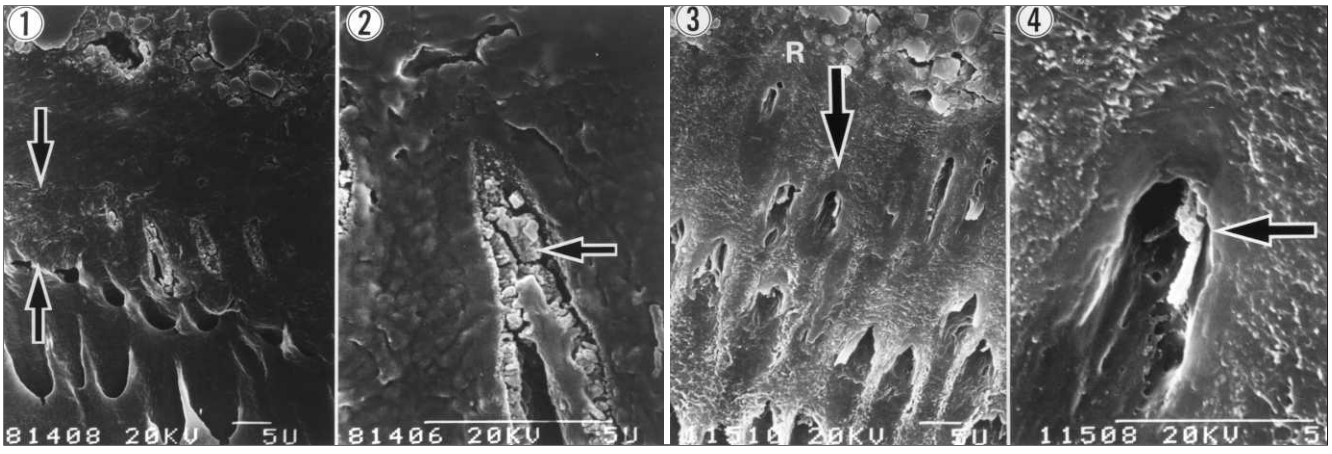


Figure 4. *SEM images of dry caries-affected dentin bonded with SB (1) Dry caries-affected dentin sectioned through the bonded interface and polished. Relatively thick (ca. 10 µm) hybrid layers (arrows) were seen. Abbreviations are the same as in Figure 2. (2) Higher magnification of the polished interface. Mineralized casts (arrow) were seen in the dentinal tubules. (3) Dry caries-affected dentin bonded with SB, sectioned, polished, and etched with acid/base treatment. The thick hybrid layer (arrow) was more susceptible to acid/base challenge than hybrid layers created in moist caries-affected dentin. R = resin. (4) Higher magnification of the etched interface of Figure 4-3. A small resin tag (arrow) was seen in the lumen.*

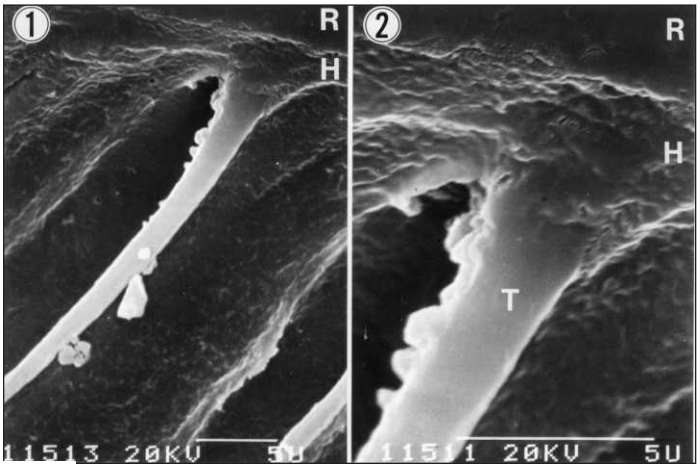


Figure 5. *SEM images of dry normal dentin bonded with SB. Abbreviations are the same as in Figure 2. (1) Dry normal dentin bonded with SB, sectioned, polished, and etched with acid/base treatment. The hybrid layer (H) was thinner than those obtained in moist normal dentin. (2) Higher magnification of the hybrid layer. The hybrid layer was resistant to acid/base challenge.*

organic substances in caries-affected dentin that interfere with uniform resin permeation or with conversion of monomer to polymer. Clearly, more research is needed in this area.

The very thin hybrid layers created in caries-affected dentin by FB were probably responsible for the low tensile bond strength. The thinness of the hybrid layer is undoubtedly due to resistance of caries-affected dentin to the self-etching primer, which is less acidic than 35% phosphoric acid. Caries-affected dentin contains deposits of whitlockite in the dentinal tubules (Daculsi & others, 1979). When smear layers are created on caries-affected dentin, it is likely that they include acid-resistant crystals and extrinsic proteins that have permeated into the mineral phase during cycles of demineralization. These smear layers may be more resistant to the action of self-etching primers than are smear layers formed from normal dentin. If the self-etching/self-priming resin cannot etch through the smear layer into the underlying intact dentin matrix, then it will only hybridize the smear layer and there will be no permeation of resin beyond the smear layer. This tends to result in low bond strengths (Gwinnett, 1993; Yoshiyama & others, 1996). It is clear that much more research is required to optimize the use of self-etching/self-priming bonding systems on caries-affected dentin. Such dentin may require etching with 35% phosphoric acid prior to use of the self-etching primer. However, this may lead to overetching of normal dentin that often surrounds caries-affected dentin. Alternatively, the etching time of the self-etching primer may have to be extended from 10 seconds to 30 seconds to solubilize enough mineral to promote more resin infiltration.

In normal demineralized dentin, the spaces between the collagen fibrils are maintained by water during the moist bonding technique. Air drying causes evaporation of this water, collapse of the collagen fibril network, and less resin infiltration (Tay, Gwinnett & Wei, 1996, 1998). Whether this same sequence of events occurs in acid-etched caries-affected dentin is unclear. However, the results of this work indicate that the benefits of moist bonding can be extended to include caries-affected dentin, at least with the SB.

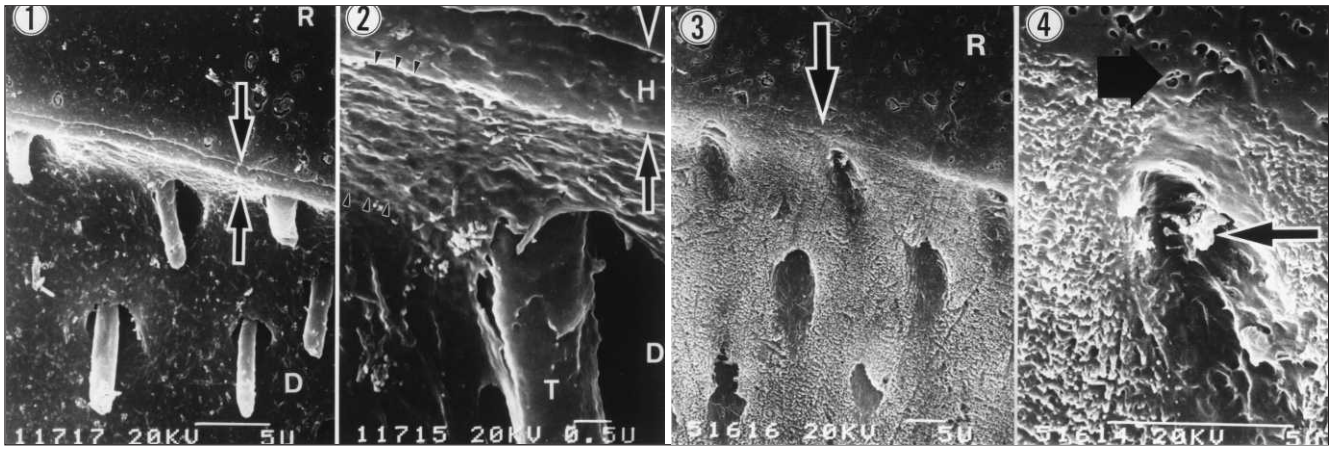


Figure 6. *SEM images of the interface created by FluoroBond (FB) in normal and caries-affected dentin after the acid-base treatment. (1) Normal dentin bonded with FB. Note that the hybrid layer (arrows) was resistant to acid and base challenge, and the thickness was about 1 µm. The resin tags were relatively short, although in this micrograph, most had been destroyed during polishing. R: bonding resin, D: dentin. (2) Higher magnification of the interface between FB and normal dentin. Below the hybrid layer (H) was a thicker acid/base resistant zone designated as a transitional layer (multiple arrows). T: resin tag. (3) SEM of the interface created by FB in caries-affected dentin. There was no detectable hybrid layer or resin tags (arrow). (4) Higher magnification of Figure 6-3 of the interface created by FB in caries-affected dentin. The interface was seen to be porous (arrow). The residual piece of resin (arrow) in the tubule was probably originally surrounded by mineral deposits that were removed when the polished surface was challenged with acid and NaOCl.*

CONCLUSIONS

Bond strengths of SB to caries-affected dentin were significantly lower ($p<0.05$) than bonds made to normal dentin using the moist bonding technique. Using the same bonding system to dry normal dentin lowered the bond strength ($p<0.05$) compared to moist normal dentin. Similarly, SB bonds made to dry caries-affected dentin were lower ($p<0.05$) than those made to moist caries-affected dentin. FB produced bond strengths that were not significantly different than those produced to dry normal dentin by SB. However, FB bonds to caries-affected dentin were significantly lower ($p<0.05$) than those made to normal dentin. The lower bond strengths produced by these bonding systems are probably due to the presence of acid-resistant mineral deposits in both dentinal tubules and in adjacent intertubular dentin. More research is needed to improve resin bonding to caries-affected dentin.

Acknowledgments

This work was supported, in part, by grant DE06427 from the National Institute of Dental and Craniofacial Research.

(Received 10 October 1998)

References

Daculsi G, Kerebel B, Le Cabellec MT & Kerebel LM (1979) Quantitative and qualitative data on arrested caries in dentine *Caries Research* **13(4)** 190-202.

Gwinnett AJ (1993) Quantitative contribution of resin infiltration/hybridation to dentin bonding *American Journal of Dentistry* **6(1)** 7-9.

Kanca J 3rd (1992) Resin bonding to wet substrate. 1. Bonding to dentin *Quintessence International* **23** 39-41.

Kanca J 3rd (1996) Wet bonding: Effect of drying time and distance *American Journal of Dentistry* **9(6)** 273-276.

Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM & Pashley DH (1995) Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesives *Journal of Dental Research* **74(10)** 1679-1688.

Pashley DH, Sano H, Ciucchi B, Yoshiyama M & Carvalho RM (1995) Adhesion testing of dentin bonding agents: a review *Dental Materials* **11(2)** 117-125.

Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R & Pashley DH (1994) Relationship between surface area for adhesion and tensile bond strengths–Evaluation of a microtensile bond test *Dental Materials* **10** 236-240.

Tay FR, Gwinnett JA & Wei SH (1996) Micromorphological spectrum from overdrying to overwetting acid-conditioned dentin in water-free acetone-based, single-bottle primer/adhesives *Dental Materials* **12(4)** 236-244.

Tay FR, Gwinnett JA, Wei SH (1998) Relation between water content in acetone/alcohol-based primer and interfacial ultrastructure *Journal of Dentistry* **26(2)** 147-156.

Yoshiyama M, Matsuo T, Ebisu S & Pashley D (1998) Regional bond strengths of self-etching/self-priming adhesive systems *Journal of Dentistry* **26(7)** 609-616.

Yoshiyama M, Sano H, Ebisu S, Tagami J, Ciucchi B, Carvalho RM, Johnson MH & Pashley DH (1996) Regional strengths of bonding agents to cervical sclerotic root dentin *Journal of Dental Research* **75(6)** 1404-1413.

©*Operative Dentistry, 2000,* **25,** 170-176

Effect of Food-Simulating Liquids on Surface Characteristics of Composite and Polyacid-Modified Composite Restoratives

AUJ Yap • JS Low • LFKL Ong

Clinical Relevance

Silux Plus, Z100, and P50 appeared to be more susceptible to the softening effects of some food-simulating liquids which could result in increased clinical wear. The large increase in surface roughness for Dyract AP could encourage plaque accumulation *in vivo*.

SUMMARY

The chemical environment is one aspect of the oral environment that could have an appreciable influence on the *in vivo* degradation of composite resins. The effects of food-simulating liquids on the surface roughness and hardness of composite (Silux Plus, Z100, Spectrum TPH, and P50) and polyacid-modified composite resins (F2000 and Dyract AP) were thus investigated and compared. Sixty disks of each material were made. Half were used for microhardness testing and the remaining half for studying surface roughness using profilometry. Each group of 30 disks was subdivided into six groups of five and conditioned for one week as follows—Group 1 (control): air at 37°C; Group 2: distilled water at 37°C; Group 3: 0.02 N citric acid at 37°C; Group 4: 0.02 N lactic acid at 37°C Group 5: heptane at 37°C; Group 6: 50% ethanol-water solution at 37°C. Data were analyzed using one-way ANOVA and Scheffé's test at a significance level of 0.05. Results showed that the surface roughness of all restoratives evaluated was not significantly affected by food-simulating liquids. No significant change in surface hardness was noted with conditioning of Spectrum TPH, Dyract AP, and F2000 in the various food-simulating liquids. The BIS-GMA-based composites Silux Plus, Z100, and P50 appeared to be more susceptible to the softening effects of some food-simulating liquids.

National University of Singapore, 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore

Adrian UJ Yap, BDS, MSc, FRSH, assistant professor, Department of Restorative Dentistry, Faculty of Dentistry. Assistant director, Center for Biomedical Materials Applications and Technology

YS Low, National Science Research Project participant, Raffles Junior College

Lucille FKL Ong, research engineer, Center for Biomedical Materials Applications and Technology

INTRODUCTION

The clinical usage of composite and polyacid-modified composite restoratives has increased substantially over the last few years due to improvements in formulation, simplification of bonding techniques, and increased aesthetic demands by patients. Composites are recommended for use in restoration of all cavity classes in anterior and posterior teeth, while polyacid-modified composites are usually indicated in nonstress-bearing areas like Class IIIs and Vs.

The physical properties of composites are dependent upon the nature of the resin matrix, filler particles, and resin-filler interface and are influenced by the chemical environment. The resin matrix becomes softened with

exposure to certain food constituents (Wu & McKinney, 1982) and plaque organic acids (Asmussen, 1984). Composites have also been shown to leach filler constituents when stored in distilled water (Söderholm & others, 1984; Øysaed & Ruyter, 1986). In addition, when composites are soaked in oral fluids, disintegration of the silane-coupling agent at the resin-filler interface occurs (Roulet & Walti, 1984). The chemical environment is, therefore, one aspect of the oral environment that could have an appreciable influence on the *in vivo* degradation of composite resins. Although much work had been done on composite restoratives, the surface degradation of polyacid-modified composite resins in chemical environments had not been widely investigated.

The objective of this study was to examine the effects of food-simulating liquids on the surface roughness and hardness of composite and polyacid-modified composite resins. The behavior of these materials when immersed in the different chemical solutions was also compared.

METHODS AND MATERIALS

Four composite resins (Silux Plus, Z100, Spectrum TPH, and P50) and two polyacid-modified composite resins (F2000 and Dyract AP) were selected for this study. The technical profiles of the materials and their manufacturers are shown in Table 1. The food-simulating liquids used for conditioning are among those recommended in the FDA guidelines (1976). Heptane simulates, for example, butter, fatty meats, and vegetable oils. Citric acid, lactic acid, and the ethanol solution simulate certain beverages (including alcoholic), vegetables, fruits, candy, and syrup. The results could, therefore, give an indication of the degree of softening in the oral environment when restoratives are exposed to different food substances.

Customized plastic molds were fabricated for the preparation of specimen disks that were 5 mm in diameter and 2 ± 0.1 mm thick. The mold was first slightly overfilled with the material under evaluation. A cellulose acetate matrix strip (Hawe-Neos Dental, Bioggio, Switzerland) was then placed over the mold and excess material was extruded by applying pressure through a glass plate of 1 mm thickness. All restoratives were then light polymerized using the Max Polymerization unit (LD Caulk/Dentsply, Milford, DE 19963) for 40 seconds through the glass plate with the exception of P50. The exposure time for P50 was 60 seconds, as recommended by the manufacturer. Sixty disks of each material were

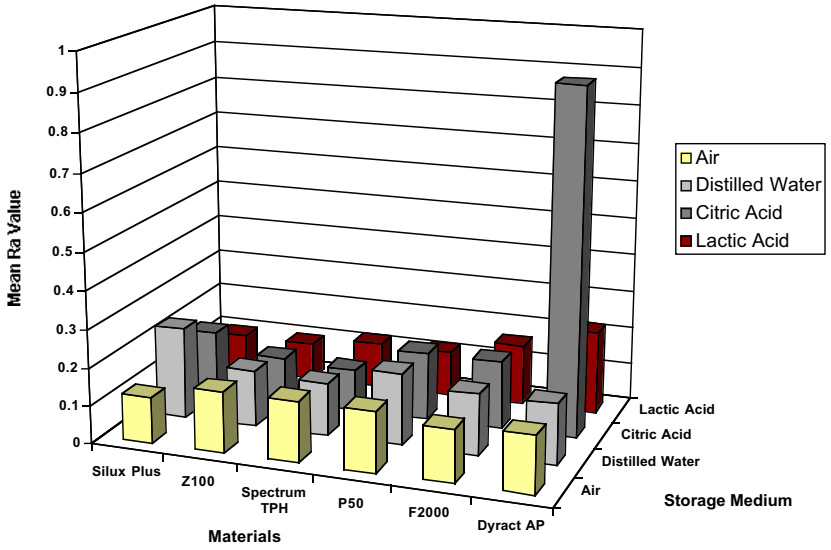
Table 1: *Technical profiles and manufacturers of the materials evaluated.*

Material	Manufacturer	Type	Resin	Filler	Filler Size (μm)	Filler Content (% by volume)	Lot No.
Silux Plus	3M Dental Products, St. Paul, MN 55144	Anterior	BisGMA TEGDMA	Silica	0.04	40	19980106
Z100	3M Dental Products, St. Paul, MN 55144	Universal	BisGMA TEGDMA	Zirconia Silica	0.04-3.5	66	19980203
Spectrum TPH	Dentsply DeTrey Konstanz, Germany	Universal	BisGMA-adduct BisEMA TEGDMA	Bariumaluminum -borosilicat Silica	0.0405	57	9970900978
P50	3M Dental Products, St. Paul, MN 55144	Posterior	BisGMA TEGDMA	Zirconia Silica	1-10	77	19970711
F2000	3M Dental Products, St. Paul, MN 55144	Anterior	CMDA GDMA	Silica	3-10	84	9801001150
Dyract AP	Dentsply DeTrey Konstanz, Germany	Universal	UMDA TCB	Strontium-fluoro-silicate glass	0.8	47	19970904

BisGMA = Bisphenol-A-glycidyl methacrylate
TEDGMA = Triethylene glycol dimethacrylate
BisGMA-adduct = Adduct of 2,2-Bis[4-2-hydroxy-3-methacryloyloxypropoxy)-phenyl]propane with hexamethylene diisocyanate
BisEMA = Ethoxylated bisphenol-A-glycidyl methacrylate
CDMA = Dimethacrylate functional oligomer derived from citric acid
GDMA = Glyceryl methacrylate
UMDA = Urethane dimethacrylate
TCB = Reaction product butane tetracarboxylic acid and HEMA

made. Half of the disks were used for microhardness testing and the remaining half for studying surface roughness. Each group of 30 disks was subdivided into six groups of five and conditioned as follows—Group 1 (control): air at 37°C; Group 2: distilled water at 37°C; Group 3: 0.02 N citric acid at 37°C; Group 4: 0.02 N lactic acid at 37°C Group 5: heptane at 37°C; Group 6: 50% ethanol-water solution at 37°C. The materials were immersed into the different food-simulating liquids immediately after light polymerization and evaluated for microhardness and surface roughness after a one-week conditioning period. The samples were lightly rinsed in water and gently dabbed dry with absorbent paper before each test measurement.

Figure 1. Mean Ra values after storage in water and weak intra-oral acids.



Microhardness Test

The Knoop hardness number (KHN) was determined for each specimen using an electronic microhardness tester (FM7; Future Tech Corp, Tokyo, Japan). Indentations were made with a 500 g load applied for 15 seconds. The mean KHN was subsequently calculated and tabulated.

Surface Roughness Evaluation

Profilometric analyses were carried out using a profilometer (Surfcom 120A; Seimitsu Corp, Tokyo, Japan) with a probe diameter of 5 µm. The profilometer was set at a vertical magnification of X2000 and horizontal magnification of X20. Ra values for each specimen were taken across the diameter over a standard length of 0.25 mm X 6. The Ra value is the arithmetic mean of the departures of the roughness profile from the mean line calculated by the computer. Specimens with more than twofold increase in Ra values after conditioning, as compared to their control, were further subjected to SEM (XL30; Philips, Shelton, CT 06484) evaluation at X2000 magnification to determine microstructural changes that may have occurred.

The data obtained were subjected to one-way ANOVA and Scheffé's test at a 0.05 significance level.

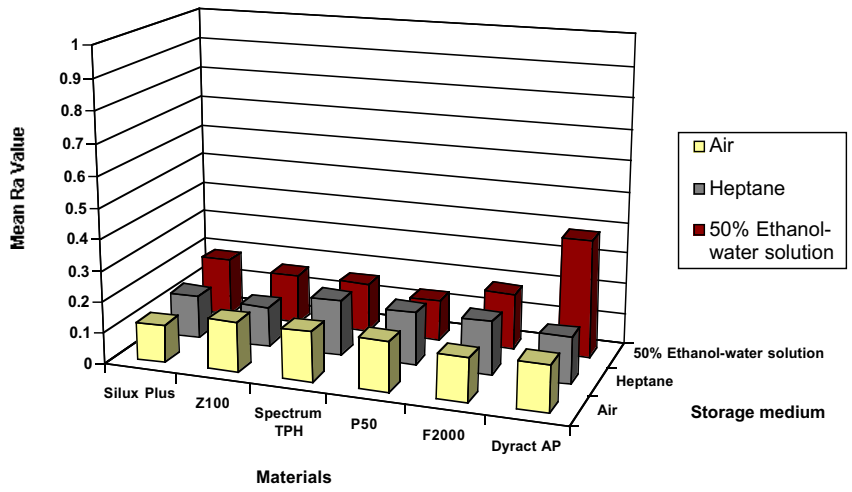
RESULTS

The mean KHN and Ra values for the different materials are shown in Table 2. The mean Ra and KHN of the materials when stored in water and weak intra-oral acids are reflected in Figures 1 and 3. Figures 2 and 4 show the mean Ra and KHN of the materials when stored in organic chemical liquids. The mean Ra and

Table 2: *Mean KHN and Ra values for the various composite restoratives (standard deviations in parenthesis).*

Medium	Silux Plus		Z100		Spectrum TPH		P-50		F2000		Dyract AP	
	Ra	KHN	Ra	KHN	Ra	KHN	Ra	KHN	Ra	KHN	Ra	KHN
Air (Control)	0.12 (0.04)	26.60 (3.61)	0.16 (0.01)	81.18 (1.65)	0.16) (0.04)	48.72 (2.59	0.16 (0.05)	76.46 (6.43)	0.14 (0.02)	52.42 (9.87)	0.15 (0.03)	31.24 (3.46)
Distilled Water	0.24 (0.07)	31.28 (1.95)	0.15 (0.05)	69.86 (6.00)	0.14 (0.04)	42.60 (2.10)	0.19 (0.05)	64.10 (11.89)	0.16 (0.02)	40.76 (7.25)	0.16 (0.02)	32.44 (3.86)
Citric Acid	0.17 (0.46)	30.62 (1.82)	0.12 (0.04)	69.76 (5.62)	0.11 (0.02)	44.58 (6.88)	0.18 (0.05)	79.98 (8.20)	0.18 (0.02)	39.74 (6.43)	0.91 (0.98)	31.76 (5.25)
Lactic Acid	0.10 (0.04)	30.52 (2.10)	0.10 (0.00)	64.48 (5.43)	0.12 (0.03)	44.26 (2.50)	0.12 (0.02)	70.10 (2.22)	0.16 (0.03)	47.16 (2.55)	0.22 (0.03)	28.76 (2.67)
Heptane	0.14 (0.04)	34.12 (2.20)	0.13 (0.02)	76.72 (7.82)	0.18 (0.06)	44.58 (11.50)	0.17 (0.04)	68.02 (4.32)	0.17 (0.05)	50.52 (15.11)	0.15 (0.02)	25.30 (7.64)
50% Ethanol-water solution	0.19 (0.12)	23.76 (2.34)	0.16 (0.03)	64.62 (3.35)	0.16 (0.01)	35.78 (3.91)	0.13 (0.02)	62.54 (4.46)	0.18 (0.02)	49.96 (2.21)	0.38 (0.25)	25.28 (3.24)

Figure 2. Mean Ra values after storage in organic chemical liquids.



KHN of materials in the control medium (air) are reflected in all figures for easy reference. Results of the statistical analysis are shown in Table 3.

Surface roughness of all materials was not significantly affected by storage in the various food-simulating liquids. Dyract AP, however, had a sixfold increase in roughness after storage in citric acid and a 2.5-fold increase in roughness with storage in 50% ethanol-water solution. These specimens were subjected to SEM evaluation. For Dyract specimens conditioned in citric acid (Figure 5A), a generalized roughening occurred with the formation of pits on the material surface. When conditioned in 50% ethanol-water solution, protrusion of the filler particles was observed (Figure 5B). A micrograph of a typical Dyract specimen stored in air (control group) is shown in Figure 5C.

No significant change in surface hardness was noted with conditioning of Spectrum TPH, Dyract AP, and F2000 in the various food-simulating liquids. There was, however, a significant decrease in hardness of Z100 after one week of conditioning in 50% ethanol-water solution and lactic acid. The KHN was 64.42 and 64.48 respectively compared to the control value of 81.18. Significant differences in KHN were also observed between P50 specimens conditioned in citric acid and ethanol-water solution. P50 specimens conditioned in citric acid (KHN 79.98) were harder than specimens stored in ethanol-water solution (KHN 62.54). Conditioning of Silux in ethanol (KHN 23.76) resulted in surface softening, which was significant when compared to hardness after storage in distilled water (KHN 31.28), citric acid (KHN 30.62), lactic acid (KHN 30.52), and heptane (KHN 34.12). Conditioning of Silux in heptane resulted in a significant increase in hardness when compared to the control (KHN 26.60).

DISCUSSION

The aesthetics and longevity of tooth-colored restoratives are highly dependent on their surface characteristics. Residual surface roughness of restorations encourages plaque accumulation, which may result in gingival inflammation, superficial staining, and secondary caries. Surface roughness of both composites and polyacid-modified composites are determined by finishing and polishing techniques (Yap, Lye & Sau, 1997) but could be affected by chemical degradation in the oral environment. Although restoratives that are cured against a matrix are not without surface imperfections, they present the smoothest surfaces possible (Stoddard & Johnson, 1991; Yap & others, 1997). This method of surface finish was used to eliminate the influence of finishing techniques on the surface roughness results.

The surface roughness of most composite and polyacid-modified composite resins was not significantly affected by conditioning in food-simulating liquids. The greatest increase of surface roughness

Table 3: *Results of statistical analysis between mediums based on Ra Values and KHN for each material.*

Materials		Differences
Silux Plus	**Ra**	NS
	KHN	Water, Citric Acid, Lactic Acid, Heptane > Ethanol-water solution Also Heptane > Control
Z100	**Ra**	NS
	KHN	Control > Ethanol-water solution and Lactic Acid
Spectrum TPH	**Ra**	NS
	KHN	NS
P-50	**Ra**	NS
	KHN	Citric Acid > Ethanol-water solution
F2000	**Ra**	NS
	KHN	NS
Dyract AP	**Ra**	NS
	KHN	NS

Results of one-way ANOVA and Scheffé's test ($p<0.05$).
> indicates statistical significance and NS indicates no statistical significance.

Figure 3. Mean KHN after storage in water and weak intra-oral acids.

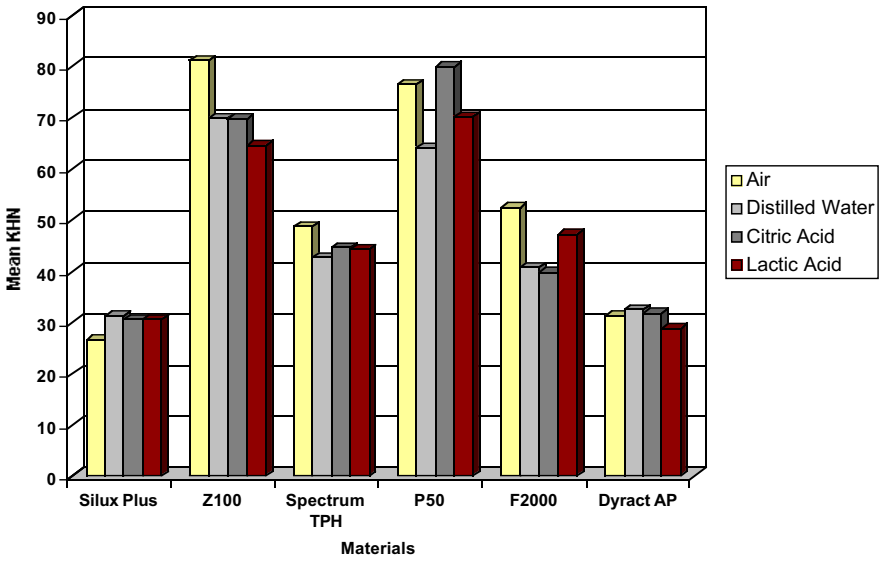
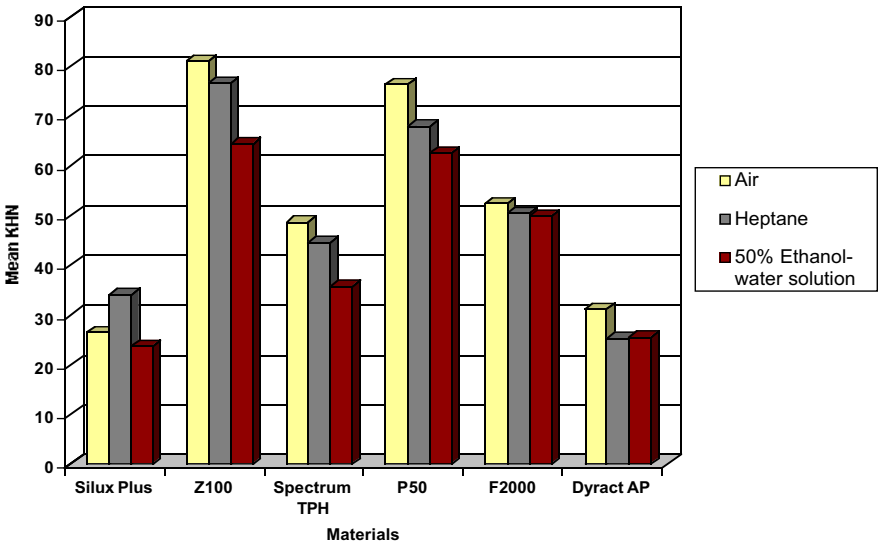


Figure 4. Mean KHN after storage in organic chemical liquids.



was noted when Dyract was conditioned in citric acid. Under SEM evaluation, a generalized roughening was observed, with the presence of numerous pits that were about the size of the filler particles (0.8 mm) in Dyract. These pits could have been caused by the loss of glass filler particles resulting from the disintegration of the coupling agent at the resin-filler interface. For specimens conditioned in 50% ethanol-water solution, the filler particles were more distinguishable from the matrix, due to chemical dissolution of the latter. A similar micro-structural change was also observed by Kao (1989) when composites were conditioned in ethanol-water solutions of different concentrations. Despite the large increase in mean surface roughness of Dyract after conditioning in citric acid and ethanol-water solution, no statistical significance was noted. This was due to wide dispersion of variance as depicted by the large standard deviation noted for Dyract specimens conditioned in these two mediums. Although microstructural changes could also have occurred with the other materials, it was not detected by profilometry. The profilometer probe diameter of 5 μm was probably not sufficiently sensitive to detect the microstructural surface changes, which are submicron in nature.

Hardness is defined as the resistance to permanent indentation or penetration. It is, however, difficult to formulate a definition that is completely acceptable, since any test method will involve complex interaction of stresses in the material being tested from the applied force. Despite this condition, the most common concept of hard and soft substances is the relative resistance they offer to indentation (Craig, 1997). Among the properties that are related to the hardness of a material are strength, proportional limit, and ductility. Hardness is a property that is used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing dental structures and materials (Anusavice, 1996). Chemical softening of restoratives may result in increased susceptibility to adhesive/abrasive, fatigue, and corrosive wear clinically. As the greatest change in hardness had been shown to occur within the first seven days (Kao, 1989) and hardness of composites is affected by conditioning for seven days (McKinney, 1985), this period of conditioning was selected for this experiment. The initial hardness after light polymerization was also recorded, but data were not used, as composites have been shown to harden post-polymerization (Chadwick & others, 1990), reflecting the progressive cross-linking reaction that occurs following initiation. The comparison of initial hardness and hardness after conditioning for a time period would thus be flawed. The best control was, therefore, specimens conditioned in air for a stipulated time period.

As the restoratives in this study were not exposed to mechanical forces, any observed changes would be from

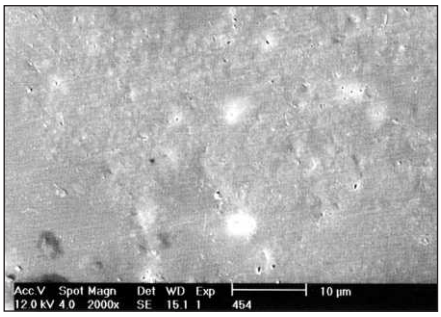


Figure 5A. *Scanning electron micrograph of Dyract after conditioning in citric acid. Note the generalized roughening and the formation of pits on the material's surface.*

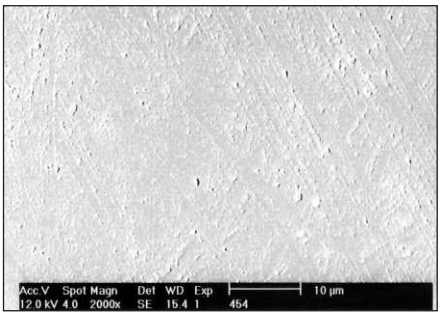


Figure 5B. *Scanning electron micrograph of Dyract after conditioning in 50% ethanol-water solution. The "protrusion" of filler particles can be observed.*

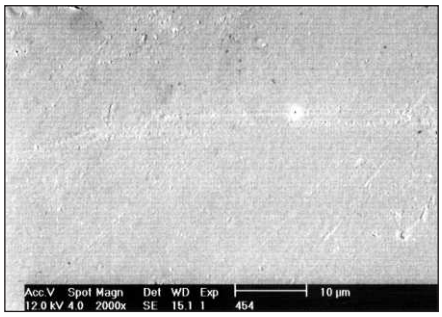


Figure 5CA. *Scanning electron micrograph of Dyract after conditioning in air (control).*

chemical dissolution. Since the sufaces of the restoratives were polymerized under pressure with an acetate strip, a matrix-rich surface was obtained. A polished surface resulting in a filler-rich surface would probably result in higher Knoop hardness values and surfaces that may be less susceptible to chemical dissolution (Kao, 1989). The surface of the material is affected before the bulk (Braden, Causton & Clarke, 1976).

The observed differences in hardness for the various materials, after conditioning in the different food-simulating liquids, may be attributed to their chemical composition and the effects of food-simulating liquids on the different chemical components. Organic liquids like heptane and ethanol-water solutions have the potential to damage the resin matrix. In addition, water and weak intraoral acids (citric and lactic acid) can damage the inorganic fillers (McKinney, 1985).

The resin matrices of Silux, Z100, and P50 are based upon BIS-GMA, whereas Spectrum is based upon the ethoxylated BIS-GMA and a BIS-GMA adduct. All these composites contain appreciable quantities of the diluent TEGDMA. The resin matrices of the polyacid-modified composite resins are reflected in Table 1. Conditioning in ethanol-water solution resulted in the softening of the surface of both composite and polyacid-modified composite resins. Irreversible processes such as the leaching of components have been shown to occur in the presence of ethanol (Lee, Greener & Menis, 1995). With the exception of F2000 and Dyract, the softening effect with ethanol-water solution was the greatest compared to conditioning in the other mediums. As the solubility parameter values for 75% to 50% aqueous ethanol solution (3 to 3.7 x 10^4 $J^{1/2}$ $m^{-3/2}$) approximated that of BIS-GMA, the softening effects on BIS-GMA-based composites were expected to be the greatest. The composite resins based on BIS-GMA (ie, Silux, Z100, and P50) appeared to be more susceptible to the detrimental effects of the ethanol solution compared to the composite based on BIS-EMA and modified BIS-GMA. This may be contributed in part to the hydrophobic nature of the ethoxylated version of BIS-GMA (BIS-EMA used in Spectrum), which does not contain unreacted hydroxyl groups on the main polymer chain (Ruyter & Nilsen, 1993).

The resin matrix of composites is known to absorb a small percentage of water, which changes the magnitude of some physical properties. Surface hardness of composites has been reported to be significantly affected by both water sorption and the contact time with the aqueous media (Hansen, 1983). This was generally the case for the composites investigated. The surface hardness of the polyacid-modified composites investigated, F2000 and Dyract, was not significantly affected by conditioning in any of the food-simulating liquids, including water. The resin matrix and filler of polyacid-modified composite resins are said to undergo an acid-base reaction after light activation and hydration. Water sorption by the polymerized resin matrix is essential for the acid-base reaction and should lead to a decrease in surface hardness as stated. As the hardness of both polyacid-modified composites was not significantly changed with conditioning in distilled water, water sorption may not have taken place within the one-week storage period.

In addition to the effects upon the resin matrix, degradation of the inorganic filler may also play a role in the reduction of hardness. All composites leach silica as a result of stress corrosion attacks upon the glass fillers (Söderholm, 1983). Those containing zinc and barium glasses are more susceptible to aqueous attack than those containing quartz (Söderholm, 1983; Øysaed & Ruyter, 1986). Quartz fillers were, however, not used in any of the restorative systems evaluated. The leakage of filler constituents has been shown to produce cracks at the resin-filler interface (Roulet & Walti, 1984; Söderholm & others, 1984), which may result in weakening of the material. The consequences upon surface hardness are, however, not clear, although it is possible that the hardness may be affected by large-scale degradation of the resin-filler interface. Lactic acid could have degraded the zirconia silicate fillers in Z100, resulting in a significant decrease in hardness. A relatively large decrease in hardness after conditioning in lactic acid

compared with the other restoratives, was also noted with P50, where the filler was zirconia silica. Lactic acid could also have caused hydrolysis of the ester groups present in the resin matrix. This reaction is acid-catalyzed and is pH-dependent (Roberts & Caserio, 1965). A significant increase in hardness was noted with Silux specimens conditioned in heptane. Two possible explanations are that heptane reduces oxygen inhibition during postcuring and eliminates leaching out of silica and combined metal in fillers, which may occur from conditioning in aqueous solutions (Söderholm, 1983).

CONCLUSIONS

1. The surface roughness of the composites and polyacid-modified composites evaluated was not significantly affected by conditioning in any of the food-simulating liquids. A large increase in surface roughness was, however, noted for Dyract AP after conditioning in citric acid and ethanol-water solution.

2. The surface hardness of both polyacid-modified composites tested, Dyract AP and F2000, was not significantly affected by the various food-simulating liquids.

3. Spectrum TPH, a BIS-EMA-based composite, was the only composite restorative tested whose surface hardness was not significantly affected by food-simulating liquids.

4. BIS-GMA-based composites, Silux Plus, Z100, and P50, appeared to be more susceptible to the softening effects of some food-simulating liquids.

(Received 20 October 1998)

References

Anusavice KJ ed (1996) *Phillips' Science of Dental Materials* 10th edition Philadelphia: WB Saunders pp 69-71.

Asmussen E (1984) Softening of BISGMA-based polymers by ethanol and by organic acids of plaque *Scandinavian Journal of Dental Research* **92(3)** 257-261.

Braden M, Causton EE & Clarke RL (1976) Diffusion of water in composite filling materials *Journal of Dental Research* **55(5)** 730-732.

Chadwick RG, McCabe JF, Walls AW & Storer R (1990) The effect of storage media upon the surface microhardness and abrasion resistance of three composites *Dental Materials* **6(2)**123-128.

Craig RG ed (1997) *Restorative Dental Materials* 10th edition London: CV Mosby pp 86-90.

Food and Drug Administration (1976) FDA Guidelines for chemistry and technology requirements of indirect additive petitions Washington DC: FDA March 1976.

Hansen EK (1983) After-polymerization of visible light activated resins: surface hardness vs light source *Scandinavian Journal of Dental Research* **91(5)** 406-410.

Kao EC (1989) Influence of food-simulating solvents on resin composites and glass-ionomer restorative cement *Dental Materials* **5(3)** 201-208.

Lee SY, Greener EH & Menis DL (1995) Detection of leached moieties from dental composites in fluids simulating food and saliva *Dental Materials* **11(6)** 348-353.

McKinney JE (1985) Environmental damage and wear of dental composite restoratives In *Posterior Composite Resin Dental Restorative Materials* Netherlands: Peter Szulc Publishing Co pp 331-347.

Øysaed H & Ruyter IE (1986) Water sorption and filler characteristics of composites for use in posterior teeth *Journal of Dental Research* **65(11)** 1315-1318.

Roberts JD & Caserio MC (1965) Alcohols and Esters In *Basic Principles of Organic Chemistry* New York: WA Benjamin Inc pp 388-391.

Roulet JF & Walti C (1984) Influence of oral fluid on composite resin and glass-ionomer cement *Journal of Prosthetic Dentistry* **52(2)** 182-189.

Ruyter IE & Nilsen J (1993) Chemical characterization of six posterior composites *Journal of Dental Research* **72** Abstracts of Papers p 177 Abstract 588.

Sôderholm KJ (1983) Leaking of fillers in dental composites *Journal of Dental Research* **62(2)** 126-130.

Sôderholm KJ, Zigan M, Ragan M, Fischlschweiger W & Bergman M (1984) Hydrolytic degradation of dental composites *Journal of Dental Research* **63(10)** 1248-54.

Stoddard JW & Johnson GH (1991) An evaluation of polishing agents for composite resins *Journal of Prosthetic Dentistry* **65(4)** 491-495.

Wu W & McKinney JE (1982) Influence of chemicals on wear of dental composites *Journal of Dental Research* **61(10)** 1180-1183.

Yap AUJ, Lye KW & Sau CW (1997) Surface characteristics of tooth-colored restoratives polished utilizing different polishing systems *Operative Dentistry* **22(6)** 260-265.

©Operative Dentistry, 2000, **25,** 177-181

Effect of Restoration Size on Fracture Resistance of Bonded Amalgam Restorations

JS Lindemuth • MS Hagge • JS Broome

Clinical Relevance

Amalgambond-Plus (with HPA) significantly improved the fracture strength of small proximocclusal amalgam restorations, but did not improve or degrade the fracture strength of large proximocclusal amalgam restorations.

SUMMARY

The purpose of this study was to determine the effect of restoration size on the fracture strength of amalgam restorations bonded with Amalgambond Plus (with HPA). Research has shown that this adhesive is dispersed throughout the unset amalgam during condensation and that a decrease in diametral tensile strength, proportional to the amount of adhesive incorporated into the unset amalgam, has resulted. Smaller cavity preparations have a higher ratio of surface area to volume than do larger preparations, and it was anticipated that a proportionately greater amount of adhesive would be incorporated into smaller amalgam restorations.

Sixty extracted human molars were divided into four groups of 15 teeth and mounted in tray acrylic-filled PVC cylinders. Shallow approximo-occlusal channels were prepared in two groups. One group was restored with Amalgambond Plus and Tytin amalgam, the other with just Tytin amalgam. Larger proximo-occlusal preparations were made in the remaining two groups, then restored in the same fashion. Samples were stored in 37°C for at least 24 hours, then thermocycled from 5-55°C 1000 times with a one-minute dwell time. Specimens were mounted in a Universal Testing Machine, and a chisel was applied to the restorations in compression mode at a crosshead speed of 5.0 mm/minute until bulk fracture of the amalgam occurred. The results indicated no difference in bulk fracture strengths between large amalgam restorations restored with and without Amalgambond Plus. However, small amalgam restorations restored with Amalgambond Plus exhibited significantly greater ($p<0.025$) bulk fracture strengths than small amalgam restorations restored without use of the adhesive.

60th Dental Squadron, Travis Air Force Base, CA 94535

James S Lindemuth, DDS, clinical investigations officer

Mark S Hagge, DMD, associate professor of Operative Dentistry, University of the Pacific Dental School, 2215 Webster St, San Francisco, CA 94115

James C Broome, DDS, colonel, USAF, CDC, clinical dentistry flight commander, 3rd Dental Squadron, Elmerdorf Air Force Base, AK 99506

INTRODUCTION

Despite its esthetic deficiencies, dental amalgam remains the most commonly used direct restorative material for posterior teeth (Christensen, 1995). The

advantages of amalgam include a relative lack of technique sensitivity, clinical longevity, and low cost. However, since amalgam does not exhibit adhesion to tooth structure, it does not have the potential to restore the strength of the prepared tooth. Recently, adhesive amalgam techniques have been introduced in an attempt to surmount this deficiency, and Christensen (1995) reported that nearly two-thirds of surveyed dentists used amalgam bonding materials.

Current adhesive amalgam techniques utilize dentin-bonding agents to achieve attachment to tooth structure (Staninec & Holt, 1988). Typically, acids are used to decalcify the dentin surface, followed by the use of hydrophilic primers to penetrate the remaining collagen network. With subsequent adhesive application, a "hybrid layer" results, and a micromechanical bond is formed to the dentin surface (Nakabayashi, Kojima & Masuhara, 1982; Inokoshi & others, 1993). The bond to the amalgam is achieved through the use of an autopolymerizing adhesive. The adhesive mix is applied to the cavity preparation, and the amalgam is condensed onto the unset polymer. Some of the polymer is forced into the amalgam and, after setting, forms a mechanical interlock.

If the polymer remained confined to the interface, no effect on the bulk mechanical properties of the restoration would be anticipated. However, previous research has indicated that the adhesive is actually dispersed throughout the amalgam during condensation. A decrease in diametral tensile strength has been noted under these conditions with certain adhesive agents (Charlton, Murchison & Moore, 1991), and this decrease has also been shown to be proportional to the amount of adhesive incorporated (Millstein & Naguib, 1995).

Smaller cavity preparations have a higher ratio of surface area to volume than do larger preparations, and since the adhesive is applied to the preparation surface, it would be expected that a proportionately greater amount of adhesive would be incorporated into smaller amalgam restorations. Smaller adhesive amalgam restorations might therefore be more likely to fail through bulk fracture than larger ones. Several related questions result: Does incorporation of adhesive adversely affect the bulk fracture strength of amalgam restorations? If a weakening effect does occur, is it dependent upon restoration size? Is any resultant weakening offset by the bonding efficacy of the adhesive agent?

Therefore, the purpose of this study was to determine the effect of restoration size on the fracture strength of adhesively-bonded amalgam restorations.

METHODS AND MATERIALS

Sixty recently extracted human third molars of similar size (10.1 to 11.4 mm in buccolingual width) were randomly separated into two groups of 30 teeth. The roots of each tooth were notched and embedded into tray acrylic (Fastray; Harry J Bosworth Co, Skokie, IL 60076) contained within a cylinder of polyvinyl chloride (PVC) tubing one inch in outside diameter. Teeth were positioned so that their long axes were parallel to the side of the PVC cylinders.

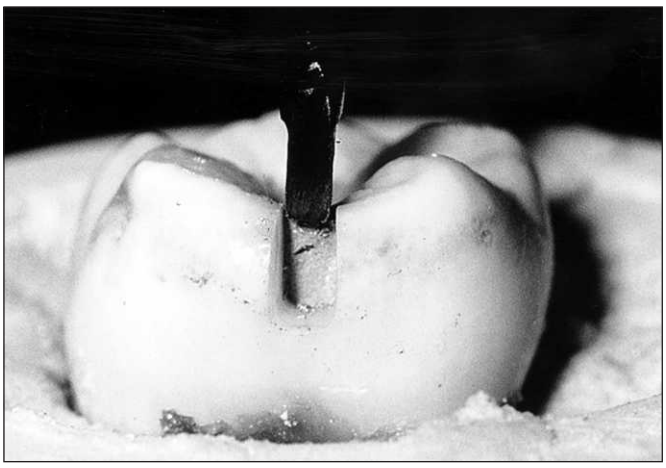


Figure 1. *Photograph of 0.8 mm-wide chisel engaged in occlusal isthmus of small restoration without Amalgambond-Plus+HPA. Fractured proximal segment has been dislodged peripherally.*

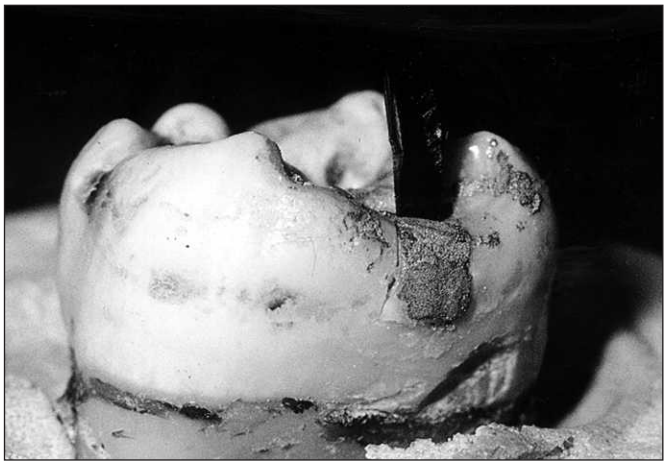


Figure 2. *Photograph of fractured small restoration with Amalgambond-Plus+HPA. Note attached peripheral enamel at linguogingival cavosurface line angle.*

A small preparation was then designed that consisted only of a simple approximo-occlusal channel incorporating a very slight occlusal convergence, but lacking a dovetail or retentive grooves as mesiodistal retentive features (Figures 1-3). The larger preparation was of classic GV Black proportions (Sigurjons, 1983), and incorporated approximal box retention grooves and an isthmus width that approached one-third the buccolingual intercuspal distance (Figures 4, 5). To maximize standardization, a "master" preparation of each design

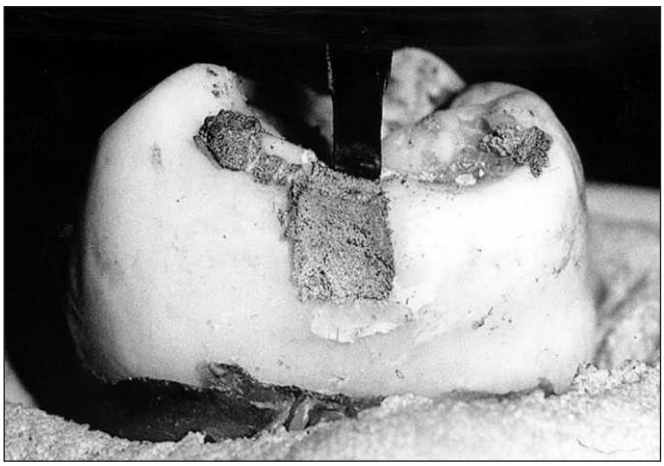


Figure 3. *Photograph of a different fractured small restoration with Amalgambond-Plus+HPA. Fractured enamel seen at the gingival cavosurface was bonded to and dislodged with the amalgam segment.*

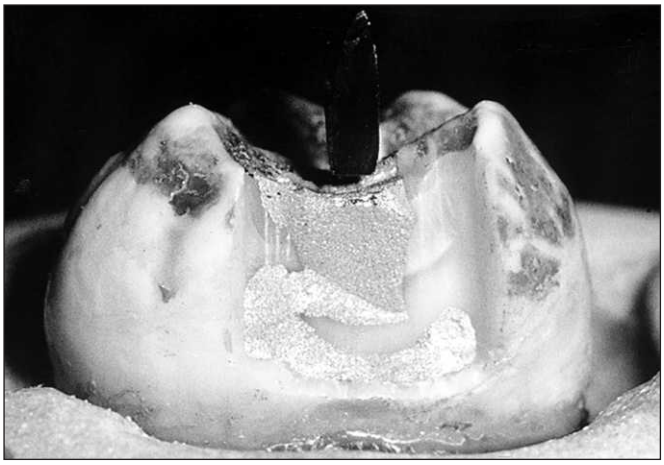


Figure 5. *Photograph of fractured large restoration with Amalgambond-Plus+HPA. The restoration has fractured obliquely, retaining amalgam within the lingual-retentive groove and axiogingival line angle.*

was made and agreed upon by both authors. Subsequently, all preparations were completed by one author (MH) and compared with the correlating master preparation by both authors before restoration.

Standardized small preparations were then made in 30 teeth using a #245 carbide bur at high speed, then gradually widened buccolingually at the occlusal isthmus to adequately permit clearance for an 0.8 mm-wide chisel. Matrices were adapted, and an amalgam adhesive treatment (Amalgambond Plus; Parkell Bio-Materials, Farmingdale, NY 11735) incorporating high-performance additive HPA (Table 1) was applied, followed by immediate placement of amalgam (Tytin; Kerr Corp, Romulus, MI 48174) using a mechanical condenser (Condensaire; Teledyne WaterPik, Ft Collins, CO 80553). This treatment was applied sequentially to each of 15 teeth. A second group of 15 teeth then had amalgam placed without pretreatment of any kind. Large

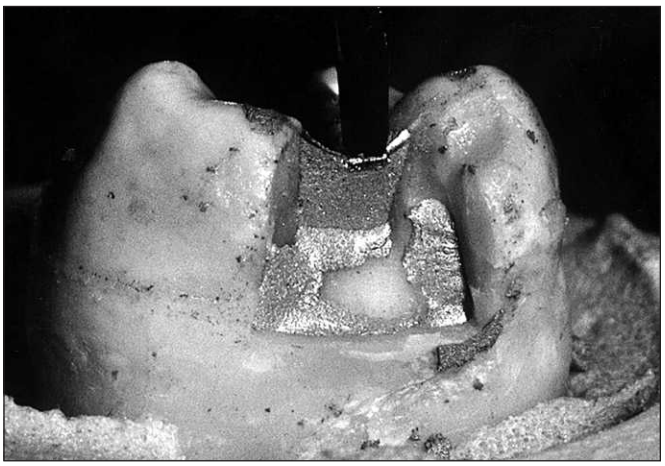


Figure 4. *Photograph of fractured large restoration with Amalgambond-Plus+HPA. The restoration has fractured obliquely, retaining amalgam within the facial and lingual retentive grooves and axiogingival line angle. Peripheral enamel shows no evidence of having bonded to the fractured amalgam segment.*

approximo-occlusal preparations described above were then made in the remaining 30 teeth using #331L and #169L carbide burs at high speed. These teeth were also divided into two groups and treated in the manner described above. Two hours following condensation, each restoration was gently scored inside the marginal ridge with a #1/4 round bur to create a flat engagement area for the chisel.

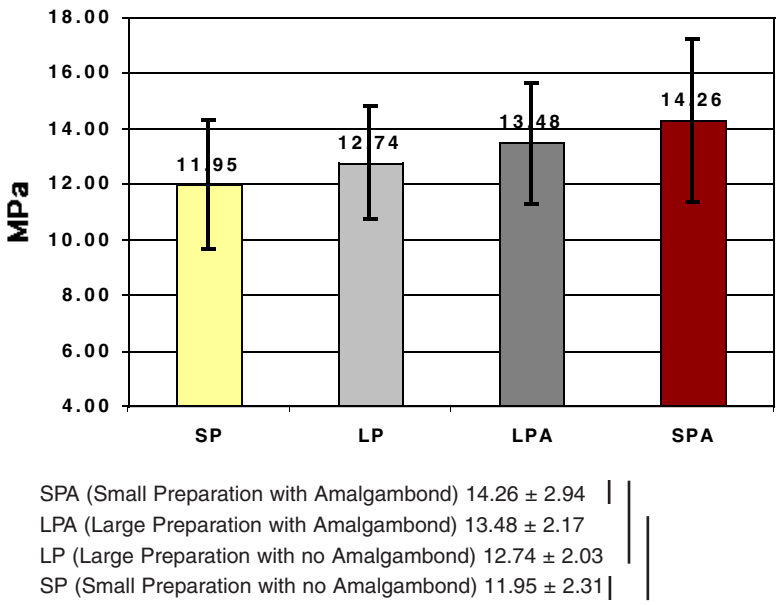
Samples were stored in distilled water at 37°C for at least 24 hours, then thermocycled from 5-55°C 1000 times with a one-minute dwell time, and returned to storage in 37°C distilled water for a period of one to three weeks. A custom fixture was fabricated from low-fusing alloy (Cerro-Bend Alloy; Cerro Metal Products, Bellefont, PA 16823) to mount the samples perpendicularly to the directed force. The chisel described previously was mounted in a Universal Testing Machine (Model 5566; Instron Corp, Canton, MA 02021) and applied to the restorations in compression mode at a crosshead speed of 5.0 mm/min until bulk fracture of the amalgam occurred. Failure strengths were recorded in kilograms (kg) and interpreted using one-way analysis of variance (ANOVA), with differences between groups determined by a post hoc Student-Newman-Keuls test. Fracture sites were also viewed under a stereomicroscope at X10 magnification.

RESULTS

Fracture strengths for each group are shown in Figure 6. Small restorations with adhesive treatment had significantly higher fracture strengths than small restorations that received no treatment ($p<0.025$), while the fracture strength of larger restorations was insignificantly affected by adhesive treatment. No other significant differences existed between groups.

Table 1.			
	Composition	**Function**	**Instructions**
Conditioner "A"	10% citric acid 3% ferric chloride	decalcifies dentin surface	apply for 30 sec., rinse, dry
Primer "AA"	HEMA	penetrates collagen	apply, leave for 30 sec., gently air dry to matte finish
Adhesive Base "B" Catalyst "C" "HPA"	MMA, 4-META TBB PMMA	micromechanical link with amalgam	mix 3 drops "B," 1 drop "C," 1 scoop HPA, brush on preparation
Means are not statistically different (p = 0.318)			

Figure 6.



Patterns of fracture remained highly consistent within groups. All small restorations without adhesive treatment cracked buccolingually through the entire depth of the restoration with the fractured segment dislodged peripherally (Figure 1). Most adhesively treated small restorations failed in a similar manner, except that small fragments of peripheral enamel remained attached (11/15) (Figures 2, 3). Other restorations in this group either fractured without attached enamel (2/15), or obliquely fractured within the amalgam, leaving some material bonded to the axial or pulpal floors (2/15). By comparison, large restorations with (Figure 4) or without (Figure 5) adhesive treatment all fractured obliquely within the amalgam, leaving some material remaining on combinations of the axial, buccal, lingual, and gingival walls. Interestingly, within the large adhesive restoration group, none of the peripherally fractured box segments contained attached enamel.

DISCUSSION

In its original design, this study attempted to use the methodology reported by Summitt, Osborne, and Burgess (1993), incorporating a flat Instron probe and a 13.5º vertical angulation of force. Unfortunately, in our laboratory, this technique consistently produced fractures of the gingival floor before the amalgam failed, and had to be modified as described above.

We found Amalgambond Plus to be highly technique sensitive in the laboratory, even though the manufacturer's instructions were closely followed. Indiscernible changes in proportion led to dramatic changes in handling characteristics, primarily in viscosity. Additionally, a noticeable loss of tactile feedback occurred during amalgam condensation in these preparations. As a result, irregularities including voids and unincorporated adhesive material were noticed occasionally at cavosurface line angles. An axial flash of adhesive was also frequently noted. The clinical relevance of these observations might be significant. For example, gingival inflammation and recurrent caries could subsequently result if these plaque-retaining defects remained undetected.

Contrary to our working hypothesis and the predictions of Charlton and others (1991), the use of Amalgambond Plus (with HPA) did not adversely affect the fracture strength of either small or large approximo-occlusal amalgam restorations. Adhesive bonding had an insignificant effect on fracture strength when used in larger restorations. However, a significant increase in strength was found in the bonded smaller restorations, suggesting that adhesive bonding of amalgam may be of significant benefit in minimally sized amalgam restorations where additional retentive features are not or cannot be placed. The most feasible explanation for this unexpected result was that the relatively larger peripheral circumference-to-volume ratio noted in smaller preparations yielded a proportionately increased bonding substrate area that effectively delayed crack propagation and subsequent fracture of the small restorations.

The larger restorations, however, incorporated retentive grooves placed within the approximal box. In an effort to absolutely minimize preparation size in the smaller samples, retentive grooves were not included in the smaller preparations, and this could have added an unintended independent variable to the study. Viewed from this perspective, our data corroborated results reported in a paper published during the data-analysis phase of our study (Della Bona & Summitt, 1998).

These authors compared Amalgambond (with HPA) and retention grooves separately and in combination in three variations of approximal-only (vertical slot) Class II amalgam preparations. They concluded that grooves and adhesive bonding were essentially equivalent in providing resistance to restoration failure.

CONCLUSION

Although the incorporation of a substantial amount of Amalgambond Plus (with HPA) enhanced the bulk fracture strength of small, minimally retentive approximo-occlusal amalgam restorations, other factors such as marginal quality, cavosurface microleakage, corrosion resistance, and occlusal wear might warrant future investigation.

Disclaimer

The views expressed in this article are those of the authors and are not to be construed as the official policy or position of the United States Air Force or the Department of Defense.

(Received 19 November 1998)

References

Charlton DG, Murchison DF & Moore BK (1991) Incorporation of adhesive liners in amalgam: effect on compressive strength and creep *American Journal of Dentistry* **4(4)** 184-188.

Christensen GJ (1995) Product use survey-1995 *CRA Newsletter* **19** 1-4.

Della Bona A & Summitt JB (1998) The effect of amalgam bonding on resistance form of Class II amalgam restorations *Quintessence International* **29(2)** 95-101.

Inokoshi S, Hosoda H, Harnirattisai C & Shimada Y (1993) Interfacial structure between dentin and seven dentin bonding systems revealed using argon ion beam etching *Operative Dentistry* **18(1)** 8-16.

Millstein PL & Naguib GH (1995) Effects of two resin adhesives on mechanical properties of set amalgam *Journal of Prosthetic Dentistry* **74(1)** 106-109.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16(3)** 265-273.

Sigurjons H (1983) "Extension for prevention": historical development and current status of G V Black's concept *Operative Dentistry* **8(2)** 57-63.

Staninec M & Holt M (1988) Bonding of amalgam to tooth structure: tensile adhesion and microleakage tests *Journal of Prosthetic Dentistry* **59(4)** 397-402.

Summitt JB, Osborne JW & Burgess JO (1993) Effect of grooves on resistance/retention form of Class II approximal slot amalgam restorations *Operative Dentistry* **18(5)** 209-213.

©*Operative Dentistry, 2000,* **25,** 182-185

Edge-Bevel Fracture Resistance of Three Direct-Filling Materials

RJ Hoard • FC Eichmiller
EE Parry • AA Giuseppetti

Clinical Relevance

The "edge strength" fracture resistance of a hand-consolidated (condensed) experimental silver filling material is similar to that of dispersed-phase amalgam and should perform clinically in a manner comparable to amalgam at cavosurface margins.

SUMMARY

Edge strength is defined in this study as the resistance to fracture of the beveled extension normally located at the cavosurface margin of a dental restoration. The edge strength of direct-filling alloy restorations plays an important role in maintaining the integrity of margins at tooth-alloy interfaces during functional loading. The purpose of this study was to determine the relative strength of an experimental consolidated silver material in comparison to other direct filling materials. The method used was designed as a simulation for relative edge-strength clinical properties.

Stainless steel dies were formed from disks 5 mm thick, each with a centered hole tapered (1/48) toward the bottom side of the disk. A 41° bevel, 0.5 mm wide as viewed from above, was placed on the top-side of the disk. Dispersalloy (D) or Unison (U) amalgam, Z-100 composite (C), hand-consolidated silver powder (HAg), or pneumatically consolidated silver powder (PAg) was used to fill the die opening. Excess was polished from both sides of the disk with 600-grit abrasive paper. The sample was loaded from the beveled side with a 3 mm-in-diameter flat-ended plunger at a rate of 1.0 mm/minute until failure. Failure load and total energy to failure were recorded and compared. Tukey's multiple comparison test ($p<0.05$) ranked the materials (U)>(HAg)>(D)> (PAg)>(C) for fracture strength and (HAg)>(D)> (U)>(PAg)>(C) for fracture energy.

American Dental Association Health Foundation, Paffenbarger Research Center, National Institute of Standards and Technology, Gaithersburg, MD 20899 USA

Richard J Hoard, DDS, clinical professor, Division of Restorative Dentistry, UCLA School of Dentistry, Los Angeles, CA 90095

Frederick C Eichmiller, DDS, director

Edward E Parry, senior dental lab technician

Anthony A Giuseppetti, AA, laboratory technician

INTRODUCTION

A material consisting of consolidated silver powder and mixtures of silver and intermetallic powders has been proposed and studied as a possible direct dental filling material. This material achieves strength by deforming and cold welding together the individual powder particles during mechanical consolidation with dental instruments

(Dariel & others, 1995; Ratzker & others, 1995; Eichmiller & others, 1996). To facilitate this cold welding, the surface contaminants are removed from the powder particles with a dilute acid, and the particles are cold-welded while submersed in the acid in a process called "acid-assisted consolidation." Studies have shown that this process can achieve flexural strengths equivalent to or exceeding those of conventional dental amalgams (Dariel & others, 1996).

One of the most common modes of failure for dental amalgam is margin failure or what has commonly been called "margin ditching." This failure is a combination of corrosion and progressive mechanical fracture of the material at the cavosurface margins (McTigue & others, 1984; Williams & Hedge, 1985). High-copper amalgams, with a greater strength and corrosion resistance, have been shown to produce less margin failure (Forsten & Kallio, 1976; Berry, Laswell & Osborne, 1980). A previous study showed that consolidated silver had a greater corrosion resistance than high-copper amalgams (Nicolae & others, 1996).

The present study attempted to compare the fracture resistance of consolidated silver to that of high-copper amalgam. Fracture resistance was measured on a specimen with a shape that would exaggerate the effect of the presence of thin, unsupported filling material as if at a beveled cavosurface margin. Previous studies have shown that the strength of the consolidated silver is related to the method and energy used to compact the material. Dental hand instruments produce adequate pressure for consolidation, but the addition of impact can significantly improve the final density and strength of the compacted material (Eichmiller & others, 1995). Most studies have used a 1.5 mm-in-diameter serrated amalgam condenser under a load of 2 kg to 3.5 kg (4.4–7.7 lb). Pneumatically assisted condensers, such as the Condensaire™ (Teledyne/Getz, Fort Collins, CO 80553) amalgam condensers and the Hollenback™ (CleveDent, Cleveland, OH 44101) gold foil condenser have been used to assist in the consolidation of this material. During consolidation, the individual silver particles undergo deformation, producing strain hardening in the consolidated mass. This strain hardening could lead to embrittlement of the material and less resistance to edge fracture. This study compared the relative edge strength of samples made from silver powder consolidated by hand instruments to those consolidated with the Hollenback pneumatically-assisted instrument in an attempt to determine if the additional strain hardening produced with the added impact imparted by the Hollenback instrument would affect the edge failure of the alloy.

METHODS AND MATERIALS

The silver powder used in this study was chemically precipitated at the laboratories of the National Institute of Standards and Technology. After precipitation, the powder was dried and sieved through a 200-mesh sieve to remove large agglomerates. The sieved powder was then annealed for two hours at 450°C before being activated in acid and consolidated. Activation of the powder was accomplished by submersing approximately 1 g in 350 mL of an aqueous solution of fluoroboric acid, volume fraction = 10% as diluted from a 48% by mass starting acid, in a glass beaker containing a slowly rotating overhead stirring paddle. The solution was stirred for one minute, then allowed to settle for approximately three minutes. The acid was then decanted, leaving the settled powder as a wet paste. The powder paste was then rinsed in a second solution by adding 500 mL of fluoroboric acid, volume fraction = 2%, and slowly stirring for approximately five seconds. The powder was once again allowed to settle, and most of the excess solution was decanted. The remaining slurry was then transferred by use of an amalgam carrier to the specimen mold for consolidation.

Table 1: *Fabrication of Edge Strength Samples*

Group	Material	Consolidation Method
1. n = 10	Dispersed phase amalgam	Hand condenser
2. n = 10	Spherical amalgam	Hand condenser
3. n = 9	Hybrid composite	Spatula and light-cure
4. n = 10	Silver powder	Hand condenser
5. n = 8	Silver powder	Pneumatic condenser

The number of acceptable samples, material, and condensation method used to fabricate samples.

Ten specimens were fabricated for each combination of material and consolidation instrument (Table 1) in a stainless steel mold with the dimensions shown in Figure 1. The consolidated silver specimens were fabricated by incrementally consolidating the powder/acid slurry into the mold. The mold cavity was a doubly tapered hole made through a 5 mm-thick by 19 mm-in-diameter stainless steel disk. A 41° bevel was placed at the surface of the top (small) end of the taper. A one part in 48 taper (opening downward) was designed to

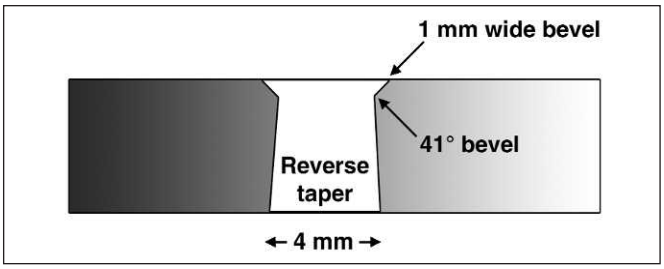


Figure 1. *A cross-section of the stainless steel sample mold and testing fixture. The reverse taper was placed to minimize friction between the sample and the mold, as the sample was loaded from the beveled end.*

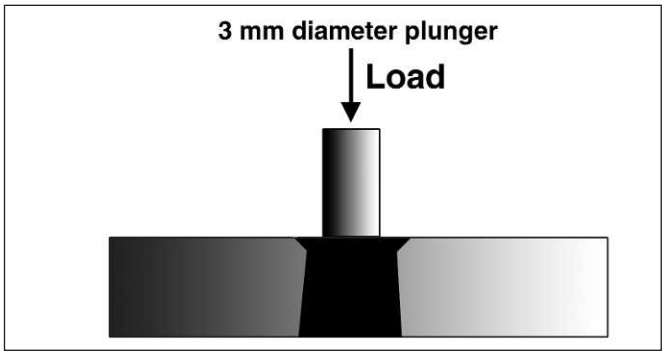


Figure 2. *Sample and loading configuration for determining edge failure strength and energy.*

minimize sliding friction as the specimen was loaded from the top beveled end and sheared to failure.

Approximately 10 increments of the silver powder were used to fill the mold cavity. Each increment was consolidated on the entire surface by overlapping the plugger contact on each increment. The alloy was consolidated with either a 1.5 mm-in-diameter serrated amalgam condenser or a Hollenback pneumatic condenser with the 1.5 mm-in-diameter serrated amalgam condensing tip fitted to the instrument. All condensation was done by hand on a load table at an instrument load ranging from 2 kg to 3.5 kg (4.4 to 7.7 lb). The mold was overfilled and the excess removed by polishing with water-cooled silicon carbide abrasive paper to a finish of 600 grit. Amalgam specimens were consolidated by using only hand condensation and were allowed to set overnight before polishing. Composite specimens were filled with a spatula and light cured for one minute under a Mylar matrix before polishing. All specimens were stored for 24 hours at room temperature before testing.

Testing was done by loading the specimen from the top, beveled end with a 3 mm-in-diameter flat plunger (Figure 2). Loading was done on an Instron Universal Testing Machine (Instron Corp, Canton, MA 02021) at a crosshead speed of 1 mm per minute, recording both load and displacement. The maximum failure load, as well as the energy to failure (area under the load-displacement curve), were determined from the load-displacement curve for each specimen. Fractures were observed under visible microscopy; one composite and two pneumatically consolidated specimens with obvious internal voids or flaws were excluded from the analysis. The failure loads and energies were subjected to analysis of variance and Tukey's multiple comparison tests at $p<0.05$.

Table 2: *Edge Fracture Load and Energy*

Material	Failure Load (N)	Failure Energy (J)
Spherical Amalgam	477 ± 107 a	0.055 ± 0.027 b
Hand Consolidated Ag	377 ± 58 b	0.144 ± 0.033 a
Dispersed Phase Amalgam	372 ± 74 b	0.073 ± 0.034 b
Pneumatically Consolidated Ag	227 ± 51 c	0.055 ± 0.014 b
Composite	147 ± 32 c	0.015 ± 0.012 c

The mean and standard deviations (±) for edge fracture and load energy.
The small letters indicate statistical grouping using Tukey's multile comparison at $p<0.05$.

RESULTS

The means and standard deviations for both failure loads and energies are given in Table 2. Load-displacement curves for these materials indicated that both amalgams and the composite fractured in a brittle mode with no indication of plastic deformation. The hand-consolidated silver had an ultimate failure load similar to the dispersed-phase amalgam. However, the load-displacement curve for this material indicated some plastic deformation before failure and a lower elastic modulus. Plastic deformation was also observed for the pneumatically consolidated silver, but to a much smaller degree. Examples of the load deformation curves are given in Figure 3.

DISCUSSION

The test method used in these experiments could give only comparative load resistances and failure energies for the different materials. No attempt was made to look at the effects of corrosion or cyclic loading on these values. Such additional experiments would provide important information for relating durability properties to clinical behavior. The consolidated silver appeared to be comparable to dispersed-phase amalgam; however, the ability to undergo plastic deformation doubled the comparable amount of energy required to fracture the silver. The fractured silver specimens differed from the amalgam in that a coherent ring, corresponding to the top, beveled portion of the sample, often sheared from the remainder as a single piece. The amalgam, in contrast, fractured into many small pieces, as did the composite. The load-displacement curves for the silver also showed a definite yield point, with plastic deformation occurring beyond the yield. The ability of silver to absorb more energy and plastically deform may translate clinically into burnishing or deforming at the margins rather than fracturing and ditching.

The lower strength and fracture energies for the pneumatically-consolidated silver may be a result of the additional strain hardening caused by the repeated impact load of the instrument. The thin beveled margin underwent considerably more strain with this instrument than with hand consolidation alone. This additional strain hardening could have caused the edges to become brittle, resulting in the lower failure loads and a lower amount of plastic deformation. The densities and

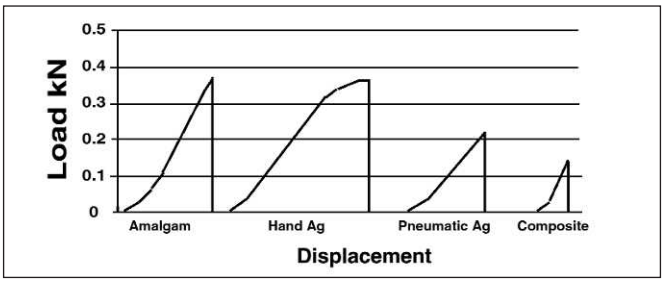


Figure 3. *Typical load-displacement curves for amalgam, hand-consolidated silver, pneumatically consolidated silver, and composite.*

fracture surfaces of the pneumatic specimens appeared to be similar on microscopic examination to the hand-consolidated silver, with no obvious flaws or defects caused by the consolidation process. The fracture resistance of the pneumatic specimens was lower than that of the hand-consolidated silver or amalgam specimens, even though the fracture energy of the pneumatic specimens was similar to that of hand-consolidated silver and amalgam. This higher fracture energy was largely due to the small amount of additional plastic deformation and lower modulus evident in the load-displacement curves for this material. Pneumatic consolidation may not be an appropriate method to use for restorations where thin cross sections or bevels are present.

CONCLUSIONS

In vitro simulations of the "edge-strength" fracture resistance of hand-consolidated silver was similar to that of dispersed-phase amalgam, while the energy required for fracture was significantly higher. Pneumatically consolidated silver was significantly weaker in edge strength, but the energy required to fracture the edge was similar to that required to fracture spherical and dispersed-phase amalgams. These data indicate that hand-consolidated silver should perform in a manner comparable to amalgam at cavosurface margins but that pneumatic consolidation may not be appropriate where restorations have thin cross sections or bevels.

Acknowledgments

This work was supported in part by USPHS Grant P50 DE09322 to the American Dental Association Health Foundation from the National Institutes of Health-National Institute of Dental Research. It 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, the ADA Health Foundation, or that the material or equipment identified is necessarily the best available for the purpose.

(Received 20 November 1998)

References

Berry T, Laswell H & Osborne J (1980) Evaluation of margin integrity of low creep amalgam alloys *Journal of the Kentucky Dental Association* **32(1)** 15-17.

Dariel MP, Admon U, Lashmore DS, Ratzker M, Giuseppetti AA & Eichmiller FC (1995) A silver-tin alternative to dental amalgams *Journal of Materials Research* **10(3)** 505-511.

Dariel MP, Levin L, Ratzker M, Stafford GR & Eichmiller FC (1996) Cold-welding effects in silver compacts after acid-assisted consolidation In *Advances in Powder Metallurgy & Particulate Materials* 1996 (Proceedings of the 1996 World Congress on Powder Metallurgy & Particulate Materials: 1996 June 16-21, Washington, DC), edited by Cadle TM & Narasimhan KS, Princeton, NJ: *Metal Powder Industries Federation* **8** 31-40.

Eichmiller FC, Giuseppetti AA, Parry EE, Hoffman KM & Ratzker MB (1995) Consolidation conditions for a cold-welded mercury-free alloy *Northwest Dentistry* **74(5)** 21-25.

Eichmiller FC, Marjenhoff WA, Dariel MP & Lashmore DS (1996) Direct metallic amalgam alternative: a research update *MSDA Journal* **39(3)** 139-141.

Forsten L & Kallio ML (1976) Marginal fracture of dental amalgams *Scandinavian Journal of Dental Research* **84(6)** 430-433.

McTigue D, Brice C, Nanda CR & Sarkar NK (1984) The *in vivo* corrosion of Disperalloy *Journal of Oral Rehabilitation* **11(4)** 351-359.

Nicolae I, Tam L, Thorpe S & McComb D (1996) Metallographic analysis and electrochemical corrosion of mercury-free direct restorative alloys *Journal of Dental Research* **75** Abstracts of Papers p 61, Abstract 350.

Ratzker M, Dariel MP, Kelley D, Giuseppetti AA, Eichmiller FC & Lashmore DS (1995) Some mechanical properties of mercury-free metallic composite restorative materials *Journal of Dental Research* **74** Abstracts of Papers p 149. Abstract 1103.

Williams PT & Hedge GL (1985) Creep-fatigue as a possible cause of dental amalgam margin failure *Journal of Dental Research* **64** 470-475.

©*Operative Dentistry, 2000,* **25,** 186-194

The Effect of Depth of Dentin Demineralization on Bond Strengths and Morphology of the Hybrid Layer

J Perdigão • KN May, Jr
AD Wilder, Jr • M Lopes

Clinical Relevance

The utilization of different phosphoric acid gels to etch dentin results in different thicknesses of the hybrid layer. It does not, however, affect the bond strengths associated with the one-bottle adhesive systems tested when dentin is etched for 15 seconds.

SUMMARY

Previous studies have shown that different phosphoric acid-based etchants do not penetrate intertubular dentin to the same depth. The purpose of this study was to determine the effect of different phosphoric acid-based conditioners on dentin shear bond strengths of three one-bottle bonding systems and to evaluate the corresponding interfacial ultramorphology. The null hypothesis to be tested was that no correlation could be established between the depth of intertubular demineralization and dentin shear bond strengths. The labial surface of 90 bovine incisors was polished to expose middle dentin. The specimens were randomly assigned to three one-bottle adhesive systems (n=30): OptiBond SOLO, Permaquick PQ1, and Single Bond. For each adhesive system the specimens were divided into three subgroups of different silica-thickened etching gels (n=10): 37.5% phosphoric acid gel (Kerr Gel Etchant), 35% phosphoric acid gel (Ultraetch), and 35% phosphoric acid gel (Scotchbond Etching Gel). After 24 hours in water at 37°C, the specimens were thermocycled for 500 cycles in baths kept at 5°C and 55°C and the shear bond strengths measured. The data were analyzed with one-way and two-way ANOVA. Further, the adhesives were applied to 800 µm-thick bovine dentin disks (two per subgroup), which were restored with a low-viscosity composite resin. Six small dentin/resin sticks with a cross-section of 1.0 mm x 1.0 mm were obtained from each bonded disk. They were then decalcified in a buffered solution of EDTA, fixed, stained, and sectioned in 90 nanometer-thick slices to observe under the Transmission Electron Microscope (TEM). The mean shear bond strengths were not statistically different at a confidence level of 95%. When the means were pooled for dentin adhesive and for etching gel, the number of cohesive failures was greater for Permaquick PQ1 and for Ultraetch, respectively. Pearson's correlation coefficient showed no correlation between hybrid layer thickness and bond strengths. The ultramorphological observation showed that all materials penetrated the

Department of Restorative Sciences, University of Minnesota, B-450 Moos Tower, Minneapolis, MN 55455

Jorge Perdigão, DMD, MS, PhD, associate professor and director, Division of Operative Dentistry

Kenneth N May, Jr, DDS, professor

Aldridge D Wilder, Jr, DDS, professor

Manuela Lopes, BS, Escola Secundária do Entroncamento, Entroncamento, Portugal

dentin and formed a hybrid layer, regardless of the etching gel used.

INTRODUCTION

Bonding to enamel is now considered a durable and predictable clinical procedure. The acid-etch technique (Buonocore, 1955) relies on the micromechanical retention created on the enamel surface by an acidic etchant and subsequent penetration of a blend of polymerizable monomers into the interprismatic spaces to form enamel resin tags (Gwinnett & Matsui, 1967). In contrast, dentin bonding has become one of the most challenging topics of restorative dentistry. The predominantly organic content and tubular structure of dentin, the presence of odontoblastic processes within the tubules, and the fluid flow in an outward direction make bonding to dentin remarkably difficult to fulfill (Swift, Perdigão & Heymann, 1995).

Recent developments in dental bonding technology involve the simultaneous etching of enamel and dentin with acids, the total-etch technique. Dentin etching has been common in Japan since the late '70s (Fusayama & others, 1979). Despite some early concerns about the possible penetration of etchants into the dentin tubules, the interaction of the etching agents with dentin is limited to the superficial few microns (Perdigão & others, 1996a,b). Thus, it seems unlikely that the acid is directly responsible for any damage to the pulp (Lee & others, 1973; Perdigão & others, 1996a; Marshall & others, 1997).

The depth of dentin demineralization has become an important issue in dentin bonding. The incomplete penetration of the demineralized microporous collagen network could result in a delicate zone inside the hybrid layer, and between the hybrid layer and the unaltered dentin, that could be susceptible to continuous degradation (Pashley, Horner & Brewer, 1992; Sano & others, 1994). While morphological studies of etched dentin are not abundant in the literature, it has been reported that phosphoric acid etchants with similar concentrations result in different depths of dentin demineralization (Perdigão & others, 1996a,b). This discrepancy may be a consequence of thickeners and other modifiers, such as buffers, introduced by the manufacturers in the composition of their proprietary etching gels. For example, the average deepest intertubular demineralization after etching with Ultraetch (Ultradent Products, South Jordan, UT 84095), a 35% phosphoric acid gel, for 15 seconds is 1.9 µm, while Scotchbond Etching Gel (3M Dental Products, St Paul, MN 55144), also a 35% phosphoric acid gel, results in a mean intertubular demineralization of 3.0 µm (Perdigão & others, 1996a,b). Another etching gel, based on 37.5% phosphoric acid (Kerr Gel Etchant; Kerr Corp, Orange, CA 92867) has been reported to result in an average deepest intertubular demineralization depth of 5.8 µm for the same etching time (Perdigão & others, 1996b).

In view of these differences in etching depth, this study was designed to evaluate the dentin shear bond

Table 1: *Dentin Adhesives Tested*

Group	Etching Gel	Mean deepest intertubular demineralization#	Thickness of the hybrid layer✢	Adhesive (Manufacturer)	Composition	Application*
KE-OP (**)	Kerr Etchant	5.8 µm	4.3-5.4 µm	OptiBond SOLO (Kerr Corp, Orange, CA 92867	BIS-GMA, GPDM, HEMA, silica, barium glass, sodium hexaflurosilicate, ethanol	abceg
SE-OP	Scotchbrand Etching Gel	3.0 µm	2.8-3.4 µm			
UE-OP	Ultraetch	1.9 µm	2.0-2.6 µm			
KE-PQ	Kerr Etchant	5.8 µm	4.3-5.4 µm	Permaquick PQI (Ultradent Products, South Jordan, UT)	TEGDMA, Canadian balsam (tree sap), 15% HEMA, 40% filler with fluoride, ethanol	abcefg
SE-PQ	Scotchbond Etching Gel	3.0 µm	2.8-3.4 µm			
UE-PQ (**)	Ultraetch	1.9 µm	2.0-2.6 µm			
KE-SB	Kerr Etchant	5.8 µm	4.3-5.4 µm	Single Bond (3M Dental Products, St. Paul, MN 55144)	BisGMA, HEMA, dimethacrylates, polyalkenoic acid, copolymer, initiator, water, ethanol	abcddfg
SE-SB (**)	Scotchbond Etching Gel	3.0 µm	2.8-3.4 µm			
UE-SB	Ultraech	1.9 µm	2.0-2.6 µm			

* -a) Apply etchant; b) rinse etchant; c) blot dry, leave moist; d) apply adhesive; e) brush on adhesive for 15 sec.; f) gently air-dry adhesive to evaporate solvent; g) light-cure adhesive.
** - Combination recommended by the respective manufacturer.
# - From Perdigão & others, 1996ab
✢ - Values measured in the present study.

strengths (SBS) of three one-bottle dentin adhesives as a function of the extension of the demineralization of intertubular dentin. The null hypothesis to be tested was that no correlation could be established between the depth of intertubular demineralization and dentin SBS.

METHODS AND MATERIALS

Dentin Shear Bond Strengths (SBS)

Ninety bovine incisors refrigerated in a solution of 0.5% chloramine for up to one week were used in this study. The teeth were cleaned of debris and mounted in phenolic rings (Buehler, Ltd, Lake Bluff, IL 60044) with cold-cure acrylic resin (Trayresin; Dentsply/Trubyte, York, PA 17405). The labial surface of each tooth was ground with 120-grit silicon carbide paper under water to expose enamel and subsequently polished for one minute with wet 240-, and 600-grit silicon carbide abrasive paper to obtain a flat smooth dentin surface. Specimens were randomly assigned to nine treatment sequences (n=10) (Table 1), as follows:

Group 1A. Dentin was etched for 15 seconds with 37.5% phosphoric acid (KE) (Kerr Gel Etchant), and rinsed for 10 seconds with copious water. OptiBond SOLO (OP) (Kerr Corp) was applied according to the manufacturer's directions. The adhesive was brushed continuously on the dentin surface for 15 seconds and light cured for 20 seconds.

Group 1B. As in Group 1A, but dentin was etched for 15 seconds with 35% phosphoric acid (SE) (Scotchbond Etching Gel; 3M Dental Products) instead of KE.

Group 1C. As in Group 1A, but dentin was etched for 15 seconds with 35% phosphoric acid (UE) (Ultraetch; Ultradent Products), instead of KE.

Group 2A. Dentin was etched for 15 seconds with KE, and rinsed for 10 seconds with copious water. Permaquick PQ1 (PQ) (Ultradent Products) was applied directly from the syringe using an Inspiral syringe tip (Ultradent Products). It was brushed on the dentin surface for 15 seconds, gently air dried to evaporate the solvent, and light cured for 20 seconds.

Group 2B. As in Group 2A, but dentin was etched for 15 seconds with SE instead of KE.

Group 2C. As in Group 2A, but dentin was etched for 15 seconds with UE instead of KE.

Group 3A. Dentin was etched for 15 seconds with KE, and rinsed for 10 seconds with copious water. Single Bond (SB) (3M Dental Products) was applied in two consecutive coats, gently air dried to evaporate the solvent, and light cured for 10 seconds.

Group 3B. As in Group 3A, but dentin was etched for 15 seconds with SE instead of KE.

Group 3C. As in Group 3A, but dentin was etched for 15 seconds with UE instead of KE.

Application of the adhesives was done according to the manufacturers' directions. Three operators placed the materials on the randomly-assigned teeth. Composite resin (Amelogen Universal, shade A2; Ultradent Products) was condensed into a #5 gelatin capsule (Torpac Inc, Fairfield, NJ 07004), to fill two-thirds of the capsule and light cured for 80 seconds in a Triad 2000 (Dentsply/Trubyte) visible-light curing unit. Following the application of the adhesive system, a final increment of composite was inserted into the gelatin capsule, and the capsule was seated securely against the flattened dentin surface. Excess material was carefully removed around the periphery of the capsule, and the composite was light cured for a total of 80 seconds (40 seconds from two opposite directions) using a Demetron 401 curing light (Demetron/Kerr, Danbury, CT 06810). The intensity of the light was monitored with a curing radiometer (Demetron/Kerr) and remained in excess of 450 mW/cm^2. After 24 hours in distilled water at 37°C, the specimens were thermocycled for 500 cycles in baths kept at 5° and 55°C, with a dwell time of 30 seconds and a transfer time of 10 seconds between baths. SBS were measured at a crosshead speed of 5 mm/minute with an Instron Universal Testing Machine (Model 4411; Instron Corp, Canton, MA 02021), using the Testworks software (MTS Systems Co, Eden Prairie, MN 55344) to record the data. The distance from the knife-edge probe to the dentin surface was monitored using a spacer of two celluloid matrices. The results were subjected to two-way analysis of variance (ANOVA) (SBS by adhesive and etchant). The statistical analysis was carried out with the SPSS 8.0 for Windows (SPSS Inc, Chicago, IL 60611) software system. After testing, the specimens were examined with a dissecting microscope at X20 to evaluate the type of failure.

Transmission Electron Microscopy (TEM)

Each of the nine combinations of acid and adhesives was applied on eighteen 800 ± 200 µm-thick additional dentin disks (two per subgroup), which were restored with a 1.0 mm-thick layer of flowable composite resin (ÆliteFlo; Bisco Inc, Schaumburg, IL 60193) and light cured for 40 seconds. Small sticks with a cross-section of 1.0 mm x 1.0 mm were cut from the bonded dentin disks using a slow-speed Isomet diamond saw (Buehler, Ltd) under water. The sticks were then decalcified in 10% buffered EDTA for 72 hours to facilitate ultramicrotomy. After removing the sticks from the EDTA, they were immersed in 2.5% glutaraldehyde/2% paraformaldehyde in 0.1 M sodium cacodylate buffer at pH 7.4 for 12 hours at 4°C. After fixation, the bonded sticks were rinsed with 10 mL of 0.1 M sodium cacodylate buffer at pH 7.4 for two hours. The specimens were post-fixed with a solution of 2% osmium tetroxide in 0.1 M sodium cacodylate buffer for one hour and washed in 0.1 M sodium cacodylate for one

Table 2: *Mean shear bond strengths and types of failure*

Treatment Group	Shear Bond Strengths Mean ± SD (MPa)	Type of Fracture*
SE-PQ	19.3±4.2	7C 1M
UE-SB	19.0±2.5	6C 1M
UE-PQ	18.2±3.0	7C 1M
UE-OP	18.2±3.2	7C 1M
KE-PQ	18.0±1.5	6C 1M
SE-SB	17.8±4.2	5C
KE-OP	17.2±2.1	4C 1M
KE-SB	16.4±4.0	4C
SE-OP	16.0±5.4	3C

SE = Scotchbond Etching Gel; KE = Kerr Etchant; UE = Ultraetch
OP = OptiBond SOLO; PQ = Permaquick PQ1; SB = Single Bond
*C = cohesive in dentin; M = mixed

Table 3: *Pooled mean SBS and types of failure (adhesive as independent variable)*

Adhesive (n=30)	Pooled SBS±S.D. (MPa)	Cohesive failure into dentin
Permaquick PQ1	18.5±3.0	20
Single Bond	17.8±3.7	15
OptiBond SOLO	17.1±3.8	14

Means are not statistically different (p = 0.393)

Table 4: *Pooled mean SBS and types of failure (etchant as independent variable)*

Etchant (n=30)	Pooled SBS±S.D. (MPa)	Cohesive failure into dentin
Ultraetch	18.5±2.8	20
Scotchbond Etching Gel	17.7±4.7	15
Kerr Gel Etchant	17.2±2.7	14

Means are not statistically different (p = 0.318)

hour. They were then rinsed with deionized water four times, with a dwell time of five minutes, and dehydrated in ascending grades of ethanol (50% for five minutes, 70% for five minutes, 95% for five minutes, and 2 x 100% for 10 minutes each). After the final ethanol step, the specimens were immersed in propylene oxide for two periods of 10 minutes each. The specimens were then embedded in 50% propylene oxide/50% MedCast epoxy resin (Ted Pella Inc, Redding, CA 96049) in a Pelco Infiltron (Ted Pella Inc) rotator at 6 rpm. After six hours of rotation, the specimens were transferred to 100% epoxy resin at room temperature, and placed under vacuum for 12 hours to allow resin infiltration into the specimens. Specimens were oriented in rubber molds so the resin-dentin interface corresponding to the central area of the dentin disk could be exposed first for sectioning. The molds were filled with fresh MedCast epoxy resin and left in an incubator during 12 hours at 65°C. The resulting resin-embedded specimen blocks were trimmed and sectioned in an MT2-B ultramicrotome (Ivan Sorvall Inc, Newtown, CT 06470) equipped with a material-sciences type III diamond knife (Micro Star Technologies Inc, Huntsville, TX 77340). After observing semi-thin specimens stained with toluidine blue under an optical microscope, the ultrathin sections (85±10 nm-thick) were cut, mounted on 150-mesh nickel grids (Ted Pella Inc) stained with 2% uranyl acetate for 20 minutes and 3% lead citrate for 15 minutes. After drying at room temperature, the sections were analyzed under a Philips CM-12 transmission electron microscope (Philips Electronic Instruments Inc, Mahwah, NJ 07430) at an accelerating voltage of 100 kV. The thickness of the hybrid layer of each section was measured directly on the microscope monitor using a point-to-point measuring device. The correlation between mean SBS and thickness of the respective hybrid layer was then computed using a two-tailed Pearson's correlation analysis.

RESULTS

Mean SBS and modes of fracture are displayed in Tables 2-4. Statistical analysis revealed no statistically significant difference between pairs of means (p= 0.475). The number of cohesive failures in dentin increased with increasing mean SBS. Cohesive failures in dentin started for bond strengths above 17.40 MPa.

When the data were pooled for dentin adhesive (Table 3), PQ resulted in the highest mean SBS, although not statistically greater than the other two materials (p=0.393). PQ also resulted in the greatest number of cohesive failures into dentin. When the data was pooled for etchant (Table 4), UE resulted in the highest mean SBS, although not statistically greater than the other etchants (p=0.318). Additionally, UE resulted in the greatest number of cohesive failures among the three etchants.

After screening the bonded interfaces with TEM, the representative images were registered and photographed

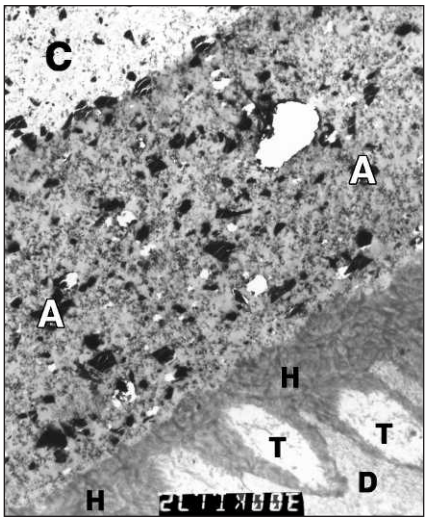


Figure 1. *Resin-dentin interface after treatment with KE-OP. The adhesive formed a 13.5-14.0 µm thick filled layer. The adhesive penetrated the demineralized collagen to form a fully-saturated hybrid layer. Note: the hybridization of the tubule wall (peritubular hybridization). Final magnification X5100.*

C-composite resin; A-adhesive; H-hybrid layer; T-resin tag; D-unaffected dentin

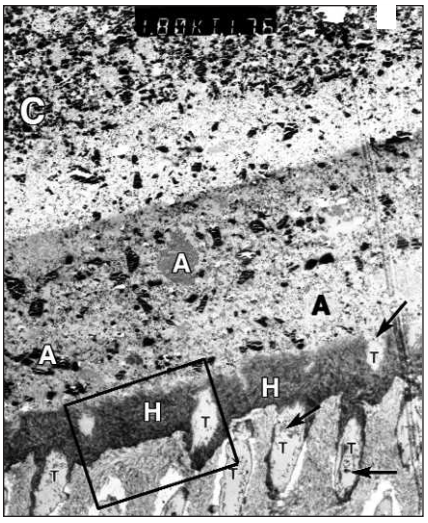


Figure 2. *Resin-dentin interface after treatment with SE-OP. The adhesive formed a 15.5 µm thick filled layer. The adhesive penetrated the demineralized collagen to form a fully-saturated 3.1 to 3.3 µm thick hybrid layer. Note: the presence of filler in the resin tags (arrows). Final magnification X3060.*

C-composite resin; A-adhesive; H-hybrid layer; T-resin tag

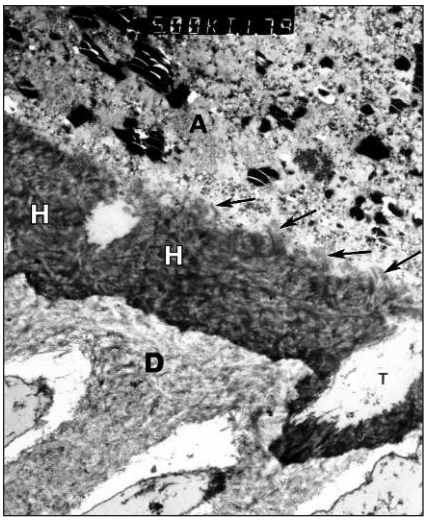


Figure 3. *A close-up of the field enclosed in the rectangle in Figure 2. Note: the shredded aspect (arrows)of the collagen fibers on the superficial part of the hybrid layer (H). The extremity of the fibers contacts directly with the adhesive and its filler particles. Final magnification X8500.*

A-adhesive; H-hybrid layer; T-resin tag; D-unaffected dentin

in negative. The penetration of the resin into the dentinal tubules, and the formation of a resin-dentin interdiffusion area, or hybrid layer (HL), were observed for all groups. The thickness of the HL at the intertubular areas varied according to the etchant used. For SE, the thickness of the HL ranged from 2.8 to 3.4 µm. For KE, the HL thickness varied from 4.3 to 5.4 µm. For UE, that thickness varied from 2.1 to 2.5 µm. No significant correlation was noted between HL thickness and mean SBS (Pearson correlation coefficient, r= -0.136, $p < 0.163$).

OP and PQ, two filled adhesives, demonstrated a very similar morphology (Figures 1-5). Both adhesives formed a thick filled layer of adhesive between the HL and the composite resin. This adhesive layer reached a thickness of 18 µm for OP and 12 µm for PQ. The mineral filler contacted the collagen fibers at the top of the HL, and, in some areas, involved the superficial loosely arranged collagen fibers (Figure 3). Filled resin tags were observed in one-third of the tubules. Regardless of the demineralization depth, both adhesives hybridized dentin, reaching the transition to the unaffected dentin without any visible gaps. The association of both SE and KE with PQ (Figure 5) resulted in a filled hybrid layer in some areas of the interface. This filled HL was not observed when PQ was combined with its proprietary etching gel (UE) (Figure 4). Additionally, lateral secondary anastomotic tubules were consistently hybridized with PQ. For OP, some areas displayed incomplete hybridization of the lateral tubule anastomoses.

SB (Figures 6, 7, and 8) hybridized dentin, reaching the transition to the unaffected dentin regardless of the demineralization depth. The lateral peritubular triangular hybridization round the tubule entrance was observed for all specimens. A layer of adhesive resin up to 8 µm-thick was observed on the top of the HL. The polyalkenoate component included in SB appeared as an electron-dense phase and interacted with the superficial 500-600 nm of the HL (Figure 6). The HL was characterized by a varying electron-density that was more pronounced immediately beneath the superficial electron-dense phase and faded gradually down toward the unaffected dentin. Above this electron-dense layer, dark fragments were observed as black "floating bubbles" dispersed within the electron-transparent polymerized adhesive (Figures 6 and 7). These small islands of electron-dense material accumulated in a long strand at the transition between the adhesive and the composite (Figures 6 and 7).

DISCUSSION

The penetration of the acids occurred primarily along the tubules (Selvig, 1968), with penetration of intertubular dentin occurring at a lower rate (Marshall & others, 1997). Acids removed the inorganic part of the dentin surface, leaving an organic backbone (Ruse & Smith, 1991). Theoretically, an acid with low molecular weight, such as nitric acid, would penetrate into

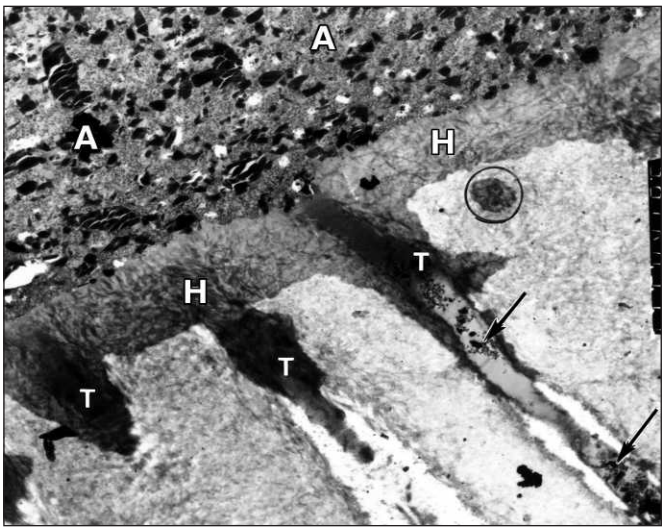


Figure 4. *Resin-dentin interface after treatment with UE-PQ. The adhesive penetrated the demineralized collagen to form a fully-saturated 2.0 to 2.6 µm thick hybrid layer. The filler penetrated into the tubules (arrows). The circle encloses a lateral secondary tubule hybridized by the adhesive. Final magnification X6477.*

A-adhesive; H-hybrid layer; T-resin tag

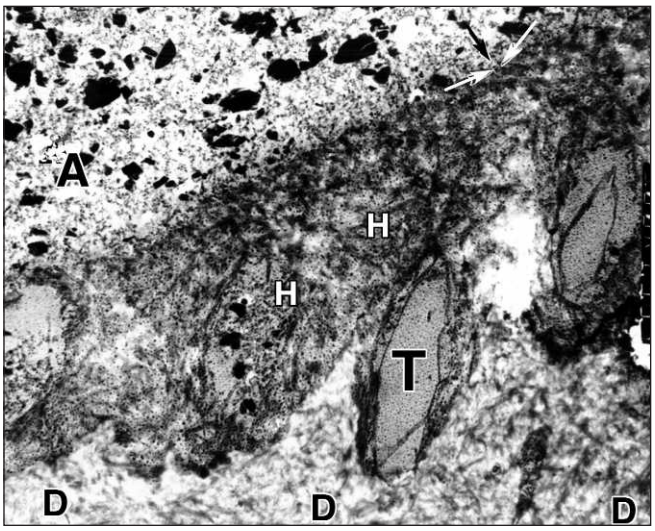


Figure 5. *Resin-dentin interface after treatment with KE-PQ. The depth of intertubular hybridization reached 5.3 µm. Small filler particles that resembled the smallest particles included in the adhesive (A) were scattered throughout the hybrid layer and transformed the resin tags into filled resin tags. The small arrows point to a microchannel between contiguous collagen fibers on the top of the hybrid layer where the particles seem to have penetrated. Final magnification X8500.*

A-adhesive; H-hybrid layer; T-resin tag; D-unaffected dentin

dentin about 89 µm if applied for 40 seconds & others, 1992). However, the interaction of the etching agents with dentin is limited by the buffering effect of hydroxyapatite and other dentin components (Wang & Hume, 1988), including the collapsed collagen, which may act as a barrier that reduces the rate of demineralization (Uno & Finger, 1996). Also, all acids currently used in dentistry are hypertonic (Pashley & others, 1992), thus drawing fluid outward, resulting in a reduction in acid penetration into dentin and a dilution of the acid itself (Perdigão & others, 1996a). Therefore, in addition to dentin having a self-defense buffering mechanism, the hypertonicity of the acids may be indirectly responsible for their penetration being less than theoretically expected.

Marshall and others (1997) have elucidated the importance of the pH with regard to the effects of acids on dentin surfaces. Etching rate penetration increases dramatically when the pH decreases. For example, UE penetrates less deeply into dentin than other products of similar concentration for the same etching time of 15 seconds (Perdigão & others, 1996a). This shallow penetration may be related to the higher pH of UE compared with other acids (Perdigão & others, 1996a). Some studies have reported that the pH is deemed essential for establishing high bond strengths to dentin. When aluminum oxalate (original pH=0.5) was buffered to pHs between 1.5 and 2.5, the highest bond strengths were obtained at higher pHs (de Araújo & Asmussen, 1989). Nevertheless, UE did not result in higher SBS than the other two acids in the present study (SE and KE). Other factors, such as the proteolytic degradation of collagen, have been reported to play a more important role than the pH (Klont, Damen & ten Cate, 1991).

Acid etching produces openings in dentin tubules and results in a network of collagen fibers separated by micropores within the intertubular dentin (Van Meerbeek & others, 1992; Pashley & others, 1993; Sano & others, 1994). Bonding to dentin is thought to rely on a micromechanical entanglement of hydrophilic resins into this demineralized, microporous dentin, thus forming a reticular intertwined hybrid tissue composed of collagen, residual mineral particles, and resin–the hybrid layer (Nakabayashi, Kojima & Masuhara, 1982; Van Meerbeek & others, 1993a,b). The thickness of this HL is commonly taken as an indicator of the depth of dentin demineralization, without taking into account the superficial area of dentin irreversibly dissolved by acid-etching (Uno & Finger, 1996; Van Meerbeek & others, 1992). In spite of the general understanding that thickness of HL is similar to demineralization depth, large variations in thickness of hybrid layers have been reported in the literature without convincing explanations offered. That variation may be a result of two factors. First, it may be related to the depth of demineralization obtained with each specific etching gel. Secondly, the HL is usually visualized under electron microscopy, which is only made possible after sectioning the resin-dentin interfaces. If the sectioning is not done perpendicularly to the bonding interface, the thickness

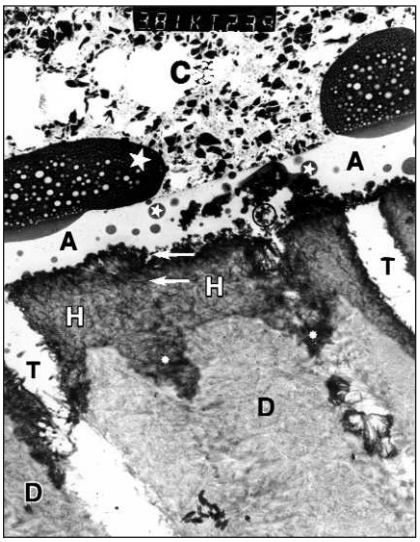


Figure 6. *Resin-dentin interface after treatment with SE-SB. The depth of intertubular reached 3.1 µm. The zones of the hybrid layer marked with an asterisk correspond to a peritubular area of a neighboring tubule that was included in the section. A 2.1 µm-thick layer of adhesive resin separates the hybrid layer from the composite resin. Note: the hybrid layer is electron-denser immediately below the adhesive. This 500-600 mm-thick dense layer is limited by two white arrows. The electron density then fades gradually down toward the center of the hybrid layer and the unaffected dentin. Electron-dense "floating bubbles" were dispersed within the polymerized adhesive (small stars). Electron dense wide islands of polyalkenoate-derived material (large stars) accumulated in a long strand at the transition between the adhesive and the composite. A small circle encloses residual silica particles left by the etchant. Final magnification X6477.*

C-composite resin; A-adhesive; H-hybrid layer; T-resin tag; D-unaffected dentin

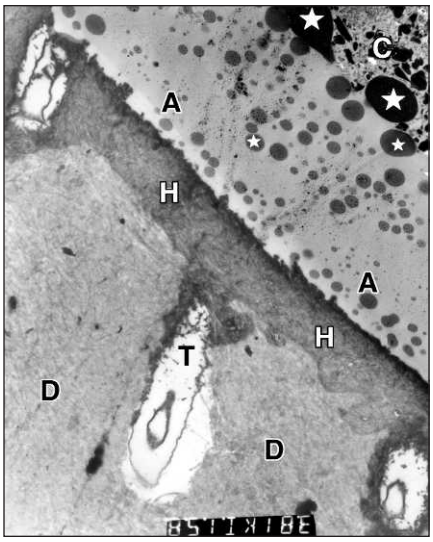


Figure 7. *Resin-dentin interface after treatment with UE-SB. The adhesive formed a 7.3 µm thick layer. The adhesive penetrated the demineralized collagen to form a fully-saturated 2.2-2.3 µm thick hybrid layer. The large stars denote the accumulation of the polyalkenoate-based substance at the transition between composite resin and adhesive. The characteristic electron dense "floating bubbles" were dispersed within the polymerized adhesive (small stars). Final magnification X6477.*

C-composite resin; A-adhesive; H-hybrid layer; T-resin tag; D-unaffected dentin

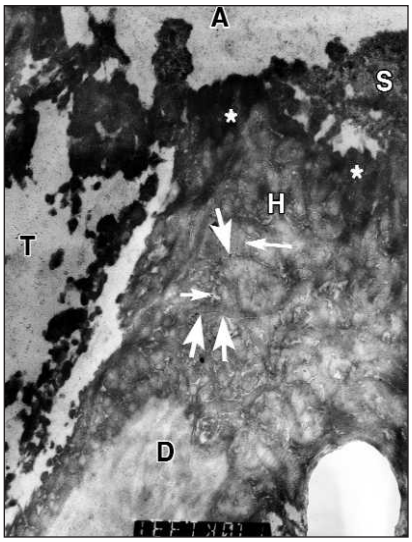


Figure 8. *Resin-dentin interface after treatment with KE-SB. This micrograph shows a high magnification of a region corresponding to a tubule entrance. The adhesive penetrated the demineralized collagen to form a fully-saturated 5.3-5.4 µm thick hybrid layer. Silica particles (S) accumulate on the hybrid layer surface. The superficial zone of the hybrid layer does not have any anatomic details (asterisk) due to the accumulation of the polyalkenoate-based electron-dense material. Note: the nano-space (small arrows) separating resin-embedded collagen which is sectioned either longitudinally or transversally. The fibers sectioned parallel to their long axis show the characteristic collagen banding (large arrows). Final magnification X28900).*

A-adhesive; H-hybrid layer; T-resin tag; D-unaffected dentin

of hybrid layer will appear thicker due to the angulation of the sections.

The thickness of the HL and its influence on bonding durability is still uncertain. Some authors have suggested that the dimensions of the HL may be taken as an indicator of the strain-absorbing capacity of the corresponding interface (Van Meerbeek & others, 1993b; Perdigão & others, 1996a). This elastic buffer could be of utmost importance for absorbing the stresses originated from composite resin polymerization shrinkage. Nevertheless, no correlation was established between thickness of the HL and bond strengths in the present study. This is in agreement with other studies (Uno & Finger, 1996; Finger & Fritz, 1996; Yoshiyama & others, 1996; Prati & others, 1998).

The bond strengths obtained in this project may be underestimated. It has been reported that there are no cohesive failures in dentin even for SBS as high as 29 MPa. If cohesive failures occur, the strength of the resin dentin interface remains undetermined (Yoshiyama & others, 1995). Cohesive failures occurring in a bonding area of 4.3 mm in diameter may not be a direct result of the effectiveness of the bond, but reflect the weakness of the thin dentin substrate. One major problem with using bovine incisors for SBS testing is that the distance from the bonding surface to the pulp chamber is thinner than with human molars (unpublished observations). Sano and others (1994) reported that bond strengths are inversely related to the bonded surface area. With small bonded surface

areas, the bonds are predominantly of the adhesive type. Recent studies have reported high bond strength values for SB (Latta & others, 1997; Swift & Bayne, 1997; Inai & others, 1998), while other studies have reported bond strengths in the same range obtained in this study (Perdigão, Ramos & Lambrechts, 1997; de Wet & others, 1998). This small discrepancy may be a result of different substrates and testing conditions. The relatively high bond strengths consistently obtained with SB may be a consequence of being a poly-alkenoic acid-based adhesive. Poly-alkenoic acid-based adhesives have been associated with resistance to degradation in a humid environment (Eliades, 1993). Additionally, there may be an intrinsic stress-relaxation capacity in Ca-polyalkenoic acid complexes (Eliades, 1993). A polyalkenoic acid-based complex has been described as forming an electron-dense material within the adhesive layer at the top area of the hybrid layer (Van Meerbeek & others, 1998). Despite clinical evidence of microleakage occurring at enamel margins when Scotchbond Multi-Purpose (3M Dental Products), a polyalkenoic-based multi-bottle bonding system, was used on maleic acid-etched enamel, the corresponding clinical retention rate at three years was unexpectedly high, which may be related to its stress-relaxation capacity (Van Meerbeek & others, 1996). Recently, it has been reported that the bond strengths associated with Scotchbond Multi-Purpose Plus increased from baseline to 10 months, which also suggested good stability of the material over time (Ario & Reistad, 1996).

For OP, most studies have reported bond strengths within the same range as the present project (de Wet & others, 1998; Wilder & others, 1998). PQ is a relatively new material without published independent studies. Data from the manufacturer suggest that PQ results in high SBS (Ultradent Products, personal communication). PQ and OP formed thick filled layers of adhesive above the hybrid layer. The formation of thick layers of adhesive resin with a low modulus of elasticity may act as stress-relaxation buffers, absorbing the tension stresses induced by the polymerization contraction of the resin composite placed over the resin (Van Meerbeek & others, 1994). For PQ, the filler penetrated the HL in some areas, when dentin was etched with KE or with SE. This HL reinforcement may have been responsible for the number of cohesive failures obtained with both combinations KE-PQ and SE-PQ.

CONCLUSIONS

The null hypothesis advanced is rejected. Different acids resulted in statistically similar bond strengths. Resin composites shrink during polymerization, causing contraction stresses of up to 7 MPa to develop within the resin (Hegdahl & Gjerdet, 1977; Davison & de Gee, 1984; Davidson, de Gee & Feilzer, 1984). It has been estimated that shear bond strengths of 17-20 MPa may be required to resist contraction forces sufficiently to produce gap-free restoration margins (Davidson & others, 1984; Feilzer, de Gee & Davidson, 1987). Therefore, the adhesives tested in this study may result in bond strengths of sufficient magnitude to counteract the contraction stresses that develop within the bonded interface. Further studies should concentrate on the effect of different etchants on the physical characteristics of the HL.

Acknowledgments

The manufacturers donated the dentin bonding systems and the composite resins tested in this project.

(Received 20 November 1998)

References

Ario PD & Reistad CA (1996) Scotchbond Multi-Purpose Plus bond stability for adhesive bonded amalgam *Journal of Dental Research* **75** Abstracts of Papers p 172 Abstract 1236.

Buonocore MG (1955) A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34** 849-853.

Davidson CL & de Gee AJ (1984) Relaxation of polymerization contraction stresses by flow in dental composites *Journal of Dental Research* **63(2)** 146-148.

Davidson CL, de Gee AJ & Feilzer A (1984) The competition between the composite-dentin bond strength and the polymerization contraction stress *Journal of Dental Research* **63 (12)**1396-1399.

de Araújo PA & Asmussen E (1989) Aluminum oxalate/glycine solutions as pretreatment in the Gluma bonding system *Scandinavian Journal of Dental Research* **97(6)** 552-558.

de Wet FA, Van der Vyer PJ, Ferreira MR & Van Rensburg JMJ (1998) One-bottle bonding systems: bond strengths and Field Emission SEM *Journal of Dental Research* **77** Abstracts of Papers p 944 Abstract 2503.

Eliades GC (1993) Dentine bonding systems In *State of the Art on Direct Posterior Filling Materials and Dentine Bonding*, edited by G Vanherle, M Degrange, & G Willems, Leuven: Van der Poorten, pp 49-74.

Feilzer A, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66** 1636-1639.

Finger WJ & Fritz U (1996) Laboratory evaluation of one-component enamel/dentin bonding agents *American Journal of Dentistry* **9(5)** 206-210.

Fusayama T, Nakamura M, Kurosaki N & Iwaku M (1979) Non-pressure adhesion of a new adhesive restorative resin *Journal of Dental Research* **58(4)** 1364-1370.

Gwinnett AJ & Matsui A (1967) A study of enamel adhesives. The physical relationship between enamel and adhesive *Archives of Oral Biology* **12(12)** 1615-1620.

Hegdahl T & Gjerdet NR (1977) Contraction stresses of composite filling materials *Acta Odontologica Scandinavica* **35(4)** 191-195.

Inai N, Kanemura N, Tagami J, Watanabe LG, Marshall SJ & Marshall GW (1998) Adhesion between collagen depleted dentin and dentin adhesives *American Journal of Dentistry* **11(3)** 123-127.

Klont B, Damen JJM, ten Cate JM (1991) Degradation of bovine incisor root collagen in an *in vitro* caries model *Archives of Oral Biology* **36(4)** 299-304.

Latta MA, Wilwerding TM, Barkmeier WW & Los SA (1997) Laboratory evaluation of one-component dental adhesive *Journal of Dental Research* **76** Abstracts of Papers p 186 Abstract 1379.

Lee HL Jr, Orlowski JA, Scheidt GC Jr & Lee JR (1973) Effects of acid etchants on dentin *Journal of Dental Research* **52(6)** 1228-1233.

Marshall GW, Inai N, Magidi I-CW, Balooch M, Kinney J, Tagami J & Marshall SJ (1997) Dentin demineralization: Effects of dentin depth, pH and different acids *Dental Materials* **13(6)** 338-343.

Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Materials Research* **16(3)** 265-273.

Pashley DH, Ciucchi B, Sano H & Horner JA (1993) Permeability of dentin to adhesive agents *Quintessence International* **24(9)** 618-631.

Pashley DH, Horner JA & Brewer PD (1992) Interactions of conditioners on the dentin surface *Operative Dentistry* Supplement **5** 137-150.

Perdigão J, Lambrechts P, van Meerbeek B, Tomé AR, Vanherle G & Lopes AB (1996a) Morphological field emission SEM study of the effect of six phosphoric acid etching agents on human dentin *Dental Materials* **12(4)** 262-271.

Perdigão J, Lambrechts P, Van Meerbeek B & Vanherle G (1996b) A FE-SEM study of the morphology of etched dentin *Journal of Dental Research* **75** Abstracts of Papers p 390 Abstract 2982.

Perdigão J, Ramos JC & Lambrechts P (1997) *In vitro* interfacial relationship between human dentin and one-bottle dental adhesives *Dental Materials* **13** 218-227.

Prati C, Chersoni S, Mongiorgi R & Pashley DH (1998) Resin-infiltrated dentin layer formation of new bonding systems *Operative Dentistry* **23(4)** 185-194.

Ruse ND & Smith DC (1991) Adhesion to bovine dentin–surface characterization *Journal of Dental Research* **70(6)** 1002-1008.

Sano H, Shono T, Sonoda H, Takatsu T, Ciucchi B, Carvalho R & Pashley DH (1994) Relationship between surface area for adhesion and tensile bond strength—evaluation of a micro-tensile bond test *Dental Materials* **10(4)** 236-240.

Selvig KA (1968) Ultrastructural changes in human dentine exposed to a weak acid *Archives of Oral Biology* **13(7)** 719-734.

Swift EJ Jr & Bayne SC (1997) Shear bond strength of a new one-bottle dentin adhesive *American Journal of Dentistry* **10(4)** 184-188.

Swift EJ Jr, Perdigão J & Heymann HO (1995) Bonding to enamel and dentin: a brief history and state of the art, 1995 *Quintessence International* **26(2)** 95-110.

Uno S & Finger WJ (1996) Effects of acidic conditioners on dentine demineralization and dimension of hybrid layers *Journal of Dentistry* **24(3)** 211-216.

Van Meerbeek B, Dhem A, Goret-Nicaise M, Braem M, Lambrechts P & Vanherle G (1993a) Comparative SEM and TEM examination of the ultrastructure of the resin-dentin interdiffusion zone *Journal of Dental Research* **72(2)** 495-501.

Van Meerbeek B, Inokoshi S, Braem M, Lambrechts P & Vanherle G (1992) Morphological aspects of the resin-dentin interdiffusion zone with different dentin adhesive systems *Journal of Dental Research* **71(8)** 1530-1540.

Van Meerbeek B, Mohrbacher H, Celis JP, Roos JR, Braem M, Lambrechts P & Vanherle G (1993b) Chemical characterization of the resin-dentin interface by micro-Raman spectroscopy *Journal of Dental Research* **72(10)** 1423-1428.

Van Meerbeek B, Peumans M, Gladys S, Braem M, Lambrechts P & Vanherle G (1996) Three-year clinical effectiveness of four total-etch dentinal adhesive systems in cervical lesions *Quintessence International* **27(11)** 775-784.

Van Meerbeek B, Peumans M, Verschueren M, Gladys S, Braem M, Lambrechts P, Vanherle G (1994) Clinical status of ten adhesive systems *Journal of Dental Research* **73(11)** 1690-1702.

Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G, Duke ES, Eick JD & Robinson SJ (1998) A TEM study of two water-based adhesive systems bonded to dry and wet dentin *Journal of Dental Research* **77(1)** 50-59.

Wang JD & Hume WR (1988) Diffusion of hydrogen ion and hydroxyl ion from various sources through dentine *International Endodontics Journal* **21(1)** 17-26.

Wilder AD Jr, Swift EJ Jr, May KN Jr & Waddell SL (1998) Bond strengths of conventional and simplified bonding systems *American Journal of Dentistry* **11(3)** 114-117.

Yoshiyama M, Carvalho R, Sano H, Horner JA, Brewer PD & Pashley DH (1995) Interfacial morphology and strength of bonds made to superficial versus deep dentin *American Journal of Dentistry* **8(6)** 297-302.

Yoshiyama M, Carvalho R, Sano H, Horner JA, Brewer PD & Pashley DH (1996) Regional bond strengths of resins to human root dentine *Journal of Dentistry* **24(6)** 435-442.

©Operative Dentistry, 2000, **25,** 195-201

The Influence of Blood Contamination on Bond Strengths Between Dentin and an Adhesive Resin Cement

T Kaneshima • H Yatani • T Kasai
EK Watanabe • A Yamashita

Clinical Relevance

Contamination by blood of any conditioned dentin surface immediately prior to bonding reduced the bond strength between adhesive resin cement and the dentin. The strength was restored at any stage of the adhesive process by washing away the blood, air-drying, and applying a self-etching primer.

SUMMARY

The objective of this study was to determine in which step of adherent surface treatments blood contamination affects the bond strength between an adhesive resin and dentin.

The coronal part of bovine incisors was polished flat to expose the dentin. The specimens were randomly divided into three groups and specific surface treatments were applied to each group: (1) a self-etching primer application (SP group); (2) 38% phosphoric acid etching followed by primer (non-HC group); (3) acid etching followed by 10% sodium hypochlorite solution application and primer (HC group). The dentin surface was contaminated by human blood before or after either step of the surface treatments. The contaminated surfaces were washed with water after 30-second exposure and air-dried. A stainless-steel rod was adhered on the dentin surface with Panavia 21 after completing the surface treatment(s), and the tensile bond strength was measured. Fifteen subgroups including control groups were tested.

If blood contamination occurred before collagen fibers were exposed by either phosphoric acid etching or self-etching primer application, the contamination presented almost no influence on bond strength. Blood contamination of the dentin surface where collagen fibers had been exposed decreased the bond strength. However, when the contaminated collagen fibers were dissolved or when the contamination occurred after the exposed collagen fibers were dissolved, the bond strength was maintained. The bond strength was markedly decreased when the contamination occurred after the primer application (Scheffé's Comparison, SP group: $p=0.0003$, non-HC group: $p<0.0001$), but was restored by reapplication of the self-etching primer.

This study revealed that the effects of blood contamination on the bond strength of adhesive resin to dentin vary greatly depending on the adherent surface conditions.

Department of Fixed Prosthodontics, Okayama University Dental School, 2-5-1 Shikata-cho, Okayama 700-8525, Japan

Takushi Kaneshima, DDS, assistant professor

Hirofumi Yatani, DDS, PhD, professor and chair

Terou Kasai, DDS, postgraduate student

Elisete Kazumi Watanabe, DDS, PhD, visiting researcher

Atsushi Yamashita, DDS, PhD, professor emeritus

INTRODUCTION

Seventeen years have passed since adhesive resin was first marketed in the dental field. Recently, several long-term clinical outcomes of prostheses cemented with adhesive resin have been investigated (Williams & others, 1989; Paszyna & others, 1990; Hussey, Pagni & Linden, 1991; Isidor & Stokholm, 1992; Hansson, 1994), and most studies have reported favorable results. In addition, a clinical epidemiologic study revealed that the use of adhesive resin as a luting cement contributed to an increase in the longevity of the prostheses when placed in the mouth (Yamashita & Tsuji, 1997). However, the study also reported some failure cases, indicating that the excellent physical properties of the adhesive resin that were obtained *in vitro* are not always attained *in vivo*.

The properties of an adhesive resin can be diminished by various intraoral factors. These factors include high humidity in the oral cavity (Xie, Powers & McGuckin, 1993; Plasmans & others, 1996), saliva or blood contamination of the adhesive areas (O'Brien & others, 1987; Krejci, Lutz & Perisic, 1992; Xie & others, 1993; Johnson & others, 1994; Powers, Finger & Xie, 1995), tissue fluid in the dentinal tubules (Nakabayashi, Ashizawa & Nakamura, 1992), temporary cement or canal sealant remnants on the adhesive areas (Tjan & Nemetz, 1992; Terada, 1993; Watanabe & others, 1997), aging of the tooth (Sheen, Wang & Tarng, 1993), and dental caries (Ehudin & Thompson, 1994). Studies of those inhibitory factors are few, compared with the wealth of basic studies on the adherent capabilities of various adhesive resins.

It has been determined that the shape and condition of collagen fibers on the dentin surfaces can affect the adhesive strength between the adhesive resin and the dentin (Nakabayashi & Takarada, 1992; Ehudin & Thompson, 1994). Blood contamination of the adhesive areas frequently occurs in the clinical environment, and it may alter the adherent capabilities of the dentin on which collagen fibers are exposed by acid etching. The objective of this study was to investigate how and at which step of the adherent surface treatment blood contamination affects the bond strength between the adhesive resin and the dentin.

METHODS AND MATERIALS

Specimen Preparation

The coronal dentin of bovine mandibular incisors, frozen immediately after extraction, was used in this experiment. After defrosting, any soft tissue around the cervical area was removed, the radicular portion was cut, and the pulp tissue removed. The vestibular area of the coronal portion was then ground flat by a diamond disk to expose the dentin. The teeth were positioned in a silicon mold, embedded in slow-curing epoxy resin (Epofix; Struers, Copenhagen, Denmark), and polymerized for eight hours at room temperature. Using an automatic polishing machine (Automet 2/Ecomet 3; Buehler Ltd, Lake Bluff, IL 60064), the specimens were polished flat using 240- and 400-grit for surface exposure, and finally 600-grit for polishing by silicon carbide waterproof abrasive paper (Buehler Ltd) under running water. A large amount of water was used to prevent epoxy resin from being impacted into the dentin surface during the grinding procedure.

Surface Treatments

The adhesive resin used was Panavia 21 (Kuraray Co, Okayama, Japan). In this experiment V-etchant, ED Primer, and 10% sodium hypochlorite solution (NeoCleaner; Neo Pharmaceutic Co, Tokyo, Japan) were used as the etching agent, dentin primer, and solvent for organic compounds, respectively. V-etchant and ED Primer are part of the Panavia 21 system/kit. V-etchant is 38% orthophosphoric acid gel, and ED Primer is a self-etching primer (pH 3) containing phosphoric acid ester monomer (10-methacryloyloxydecyl dihydrogen phosphate), HEMA (2-hydroxyethyl-methacrylate), salicylic acid derivative monomer (N-methacryloyl-5-aminosalicylic acid), and polymerization enzyme. Sodium hypochlorite solution was used to remove any exposed collagen from the dentin surface because the use of sodium hypochlorite for deproteination of the etched dentin surface has improved bond strength (Wakabayashi & others, 1994). The blood sample used as the contamination substance was obtained from one of the authors, immediately inserted in an anticoagulant solution, and frozen. The collected blood sample was used within one week.

Tensile Bond Strength Measurement

The specimens were randomly divided into three groups: (1) specimens in which a self-etching primer (ED Primer) alone was applied (SP group); (2) specimens in which phosphoric acid etching was followed by ED Primer application (non-HC group); and (3) specimens in which ED Primer was applied after phosphoric acid etching and subsequent sodium hypochlorite application (HC group). ED Primer was applied to the air-dried dentin surface for 60 seconds, and dried with a mild stream of air. Phosphoric acid and sodium hypochlorite were applied on the dentin surface for 30 and 60 seconds, respectively. They were washed away with a stream of water, and air dried thoroughly. The contamination was carried out by applying blood from the sample using a syringe, covering the exposed dentin surface before or after either step of the surface treatments. The contaminated surfaces were washed with a stream of water after 30-second exposure and then air dried. The specimens that were not contaminated were used as controls. As a result, each group

Table 1: *A list of the adherent surface treatments for measuring tensile bond strength between an adhesive resin and the dentin*

Group	Subgroup	No. of Samples	Surface treatment
SP	1 (Control)	10	SP
	2	10	BL→ SP
	3	10	SP→BL
	4	10	SP→BL→SP
non-HC	1 (Control)	10	EE→ SP
	2	10	BL→ EE→ SP
	3	10	EE→BL→ SP
	4	10	EE→ SP→ BL
	5	10	EE→ SP→ BL→SP
HC	1 (Control)	10	EE→ HC→ SP
	2	10	BL→ EE→ HC→ SP
	3	10	EE→BL→ HC→ SP
	4	10	EE→ HC→ BL SP
	5	10	EE→ HC→ SP→BL
	6	10	EE→ HC→ SP→BL→SP

EE: 38% phosphoric acid etching(30 sec)
HC: Sodium hypochlorite solution application (60 sec)
SP: self-etching primer application (60 sec)
BL: blood contamination (30 sec)

was further divided into several subgroups according to the point at which blood contamination was carried out (Table 1).

According to the manufacturer's instructions, Panavia 21 resin cement was mixed for 20 seconds and placed on the adherent surface of a 4 µm-in-diameter stainless-steel rod (SUS-304) previously sandblasted with 50 mm aluminum oxide, which was then finger pressed onto the dentin surface. Any excess of adhesive resin was carefully removed with a small brush, and Oxyguard II (present in the Panavia 21 system/kit; Kuraray Co) was immediately placed around the rod to enhance polymerization. After standing for 10 minutes at room temperature, the Oxyguard II was washed away (Figure 1) and the specimens were placed in distilled water at 37°C for 24 hours. The specimens were positioned in a jig for tensile bond strength measurement, from which values were obtained with a Universal Testing Machine (Autograph AGS-500D; Shimadzu, Kyoto, Japan) at a crosshead speed of 2 mm/minute. Ten specimens were measured in each subgroup.

Figure 1. *A specimen for measuring tensile bond strength.*

SEM Observation of the Dentin Surfaces

Specimens other than those used for bond strength tests were prepared for surface SEM analysis, using a diamond disk to shape the dentin in a 7 x 7 mm rectangular form. Polishing and adherent surface treatment were performed in the same way as for the bond strength measurement. After fixation in 2.5% glutaraldehyde for 16 hours, dehydration in ethyl alcohol, and critical point drying (JCPD-5; JEOL Ltd, Tokyo, Japan), the specimens were gold coated in an ion coater (IB-5; Eiko Engineering Co, Ibaragi, Japan) for SEM observation (DS-720; Topcon Co, Tokyo, Japan).

Statistical Analysis

A one-way analysis of variance (ANOVA) was used to analyze the data obtained. Scheffé's Comparison was employed to evaluate statistical differences in bond strength among the subgroups. The significance level was set at $\alpha = 0.10$ to avoid excess Type II error.

RESULTS

Tensile Bond Strength Measurement

The tensile bond strengths measured are shown in Figure 2. ANOVA showed that the effects of blood contamination on the tensile bond strength were significant in all three groups ($p<0.0001$). In the SP group, blood contamination before application of ED Primer did not show any influence on the bond strength. In the non-HC group, blood contamination before and after phosphoric acid etching did not significantly influence the bond strength if ED Primer application followed. In the HC group, blood contamination before phosphoric acid etching did not influence the bond strength. When

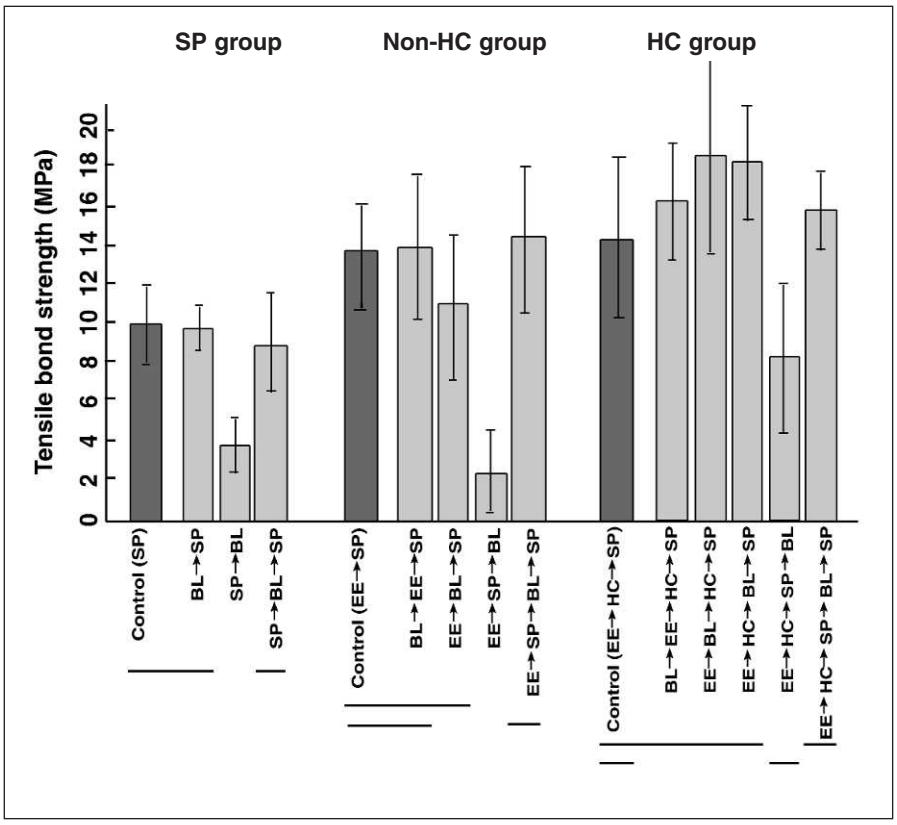


Figure 2. *Comparison of the tensile bond strength values of an adhesive resin to bovine dentin.*
EE: 38% phosphoric acid etching (30 sec)
HC: sodium hypochlorite solution application (60 sec)
SP: self-etching primer application (60 sec)
BL: blood contamination (30 sec)
(Horizontal lines on the bottom of the table show that means are not significantly different from each other.

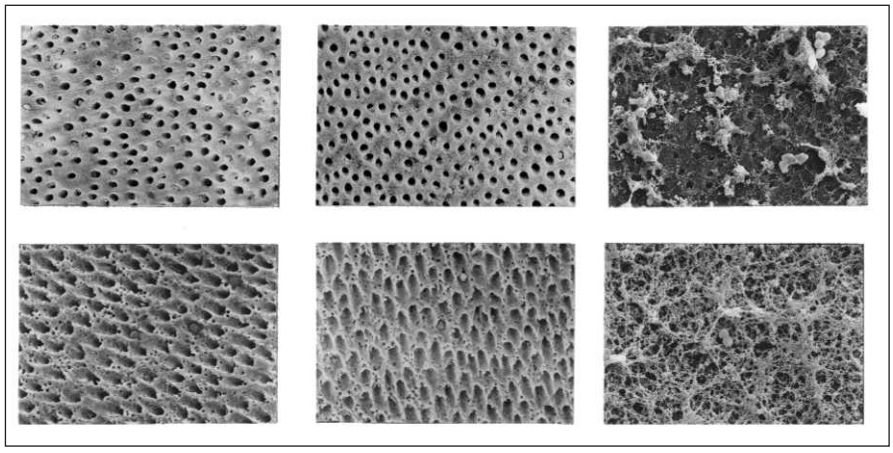


Figure 3. *SEM photographs of the dentin surfaces before applying a dentin primer. Original magnification X2000.*
Upper Row: surfaces after 38% phosphoric acid etching (non-HC group).
Lower Row: surfaces after 38% phosphoric acid etching followed by sodium hypochlorite application (HC group).
(Left): non-contaminated surfaces.
(Center): blood-contaminated surfaces (the blood was washed away with a stream of water).
(Right): blood-contaminated surfaces (the blood was air-dried only).

blood contamination occurred after phosphoric acid etching, the bond strength did not significantly change if sodium hypochlorite solution was applied to the contaminated surface. When blood contamination occurred on a dentin surface to which sodium hypochlorite solution was applied after phosphoric acid etching, the bond strength did not significantly change.

In the SP and the non-HC groups, the bond strength was markedly decreased when blood contamination occurred after ED Primer application (SP group: p=0.0003, non-HC group: p<0.0001). In the HC group the bond strength also decreased, but the decrease was not statistically significant. Return to original values occurred by reapplication of the primer in all groups.

SEM Observations of the Dentin Surfaces After Blood Contamination

SEM photographs of the dentin surfaces when the blood contamination was carried out after phosphoric acid etching (non-HC group) and after phosphoric acid etching followed by sodium hypochlorite application (HC group) are shown in Figure 3. When the blood was removed by air only, a number of residues resembling blood plasma and hemocytes were observed on the dentin surface, regardless of sodium hypochlorite solution application. When the blood was washed away with a stream of water, neither the non-HC nor the HC groups presented any differences in surface characteristics compared to each control group.

SEM photographs of the dentin surfaces after ED Primer application (non-HC and HC group) are shown in Figure 4. When the blood was removed by air only, a number of residues resembling blood plasma and a number of aggregation masses that seemed to result from the reaction of hemocytes upon the primer were observed on the dentin surface. When ED Primer was applied after the blood contamination was washed away, both regions where the primer seemed to completely penetrate and not penetrate were observed in the non-HC group. The HC group presented

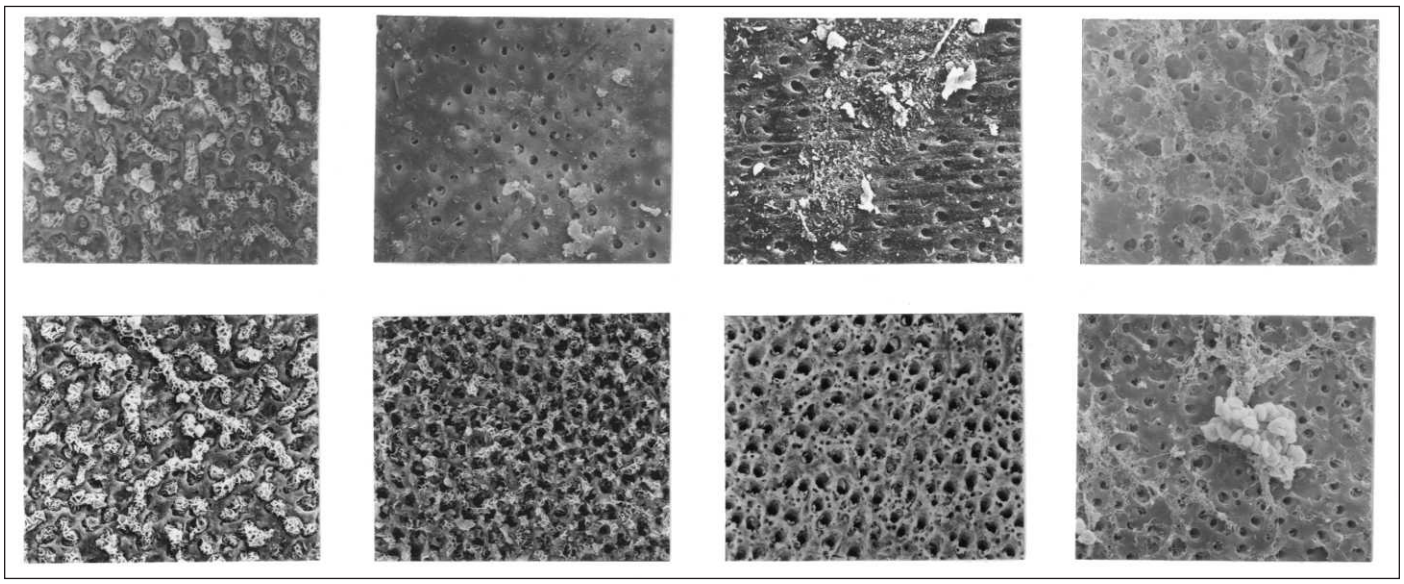


Figure 4. *SEM photographs of the dentin surfaces after applying a dentin primer. Original magnificationX2000.*
Upper Row: surfaces after 38% phosphoric acid etching followed by ED Primer application (non-HC group).
Lower Row: surfaces after 38% phosphoric acid etching and sodium hypochlorite application followed by ED Primer application (HC group).
(Left): non-contaminated surfaces.
(Left Center): blood-contaminated surfaces (the blood was applied and washed away before ED Primer application).
(Right Center): blood-contaminated surfaces (the blood was applied and washed away after ED Primer application).
(Right): blood contanimated surfaces (the blood was air-dried only before ED Primer application.)

no differences in surface characteristics compared to the control sample. When the blood contamination was carried out after ED Primer application, residues of blood contaminants were still partially observed in the non-HC group, even after the blood contamination was washed away. The HC group presented the same surface characteristics as the samples on which phosphoric acid etching was followed by sodium hypochlorite application (the control group in Figure 3).

DISCUSSION

It has been shown that the smear layer is removed from the dentin surface, exposing the dentinal collagen fibers, by phosphoric acid etching or self-etching primer application (Inagaki & others, 1989; Wang & Nakabayashi, 1991). In this study, blood contamination before acid etching or self-etching primer application did not affect the bond strength when the blood was rinsed away with water. This result indicated that the effect of blood contamination of the nontreated dentin surface was eliminated, together with the smear layer, by acid etching or self-etching primer application.

When blood contamination occurred on the dentin surface after dentinal collagen had been exposed, the bond strength was decreased. However, when blood contamination after acid etching was followed by sodium hypochlorite solution application or when the contamination occurred between acid etching and sodium hypochlorite solution application, the bond strength did not decrease. These results suggest that the inhibition of adhesion by blood contamination on the dentin is strongly associated with the superficial layer of exposed collagen, since collagen fibers exposed on the dentin surface are dissolved by the application of sodium hypochlorite solution (Wakabayashi & others, 1994). As shown in Figure 3, however, a SEM image of the sample contaminated by blood after acid etching did not present any differences in surface characteristics compared to that of the noncontaminated (control) sample when the blood was washed away with water. These SEM findings likely show that, although the relatively large blood corpuscle elements like the red globules can be completely washed away from the collagen-exposed dentin surface, blood contamination might permit some reaction between the superficial organic layer of exposed dentinal collagen and the blood protein components. This reaction might inhibit primer infiltration into the dentin or subsequent resin penetration and polymerization. In fact, when ED Primer was applied on the etched dentin surface after the blood was washed away, regions incompletely infiltrated by the primer were observed on SEM images in the non-HC group. On the other hand, SEM images in the HC group clearly showed that when the blood contamination occurred immediately after sodium hypochlorite solution application, there were no differences in surface characteristics between the contaminated samples and the controls. These findings, together

with the results of bond strength tests, suggest that dentin surfaces with exposed apatite are not influenced by blood contamination, since the blood contaminants are efficiently eliminated by rinsing and a primer can still sufficiently penetrate into the dentin.

When the blood contamination occurred after the primer application, the bond strength was significantly decreased in all three groups. In the samples of the non-HC group, residues of blood contaminants or reactants between the blood and the primer were still partially observed on the SEM image even after rinsing. The residues or reactants can become a strong mechanical inhibitor to the adhesion between the dentin and the adhesive resin cement. In the HC group, exposed dentin apatite was observed on the whole surface of the SEM image obtained from the sample in which the blood contamination was carried out after ED Primer application. The image was totally different from that of the sample contaminated before ED Primer application. This is probably because the adherent components in the primer were eliminated from the dentin surface, together with blood components, by rinsing with water, resulting in insufficient wetting abilities of the adhesive resin to the dentin (Suzuki & Nakai, 1993). However, the mechanical retention between the exposed apatite and the adhesive resin seemed to play some role for bonding the two, resulting in the nonsignificant reduction of the bond strength. When the primer was applied again after rinsing the blood contaminants with water, the bond strength was restored to that of the control group. It can be assumed from these results that regardless of collagen fiber exposure, the primer's high level of penetration into the dentin produced a primed surface that resisted the negative effects of blood contamination.

As seen in Figures 3 and 4, air blowing only spread the blood components entirely over the dentin surface, and the components covered the collagen or dentinal tubules in a net form. By applying the ED Primer to the dentin surface, the blood components cohered with each other, producing masses in several parts of the dentin surface. The contaminated layer can become a strong mechanical inhibitor to the adhesion, preventing the primer and adhesive resin from penetration and polymerization. In a preliminary experiment it was confirmed that blood contamination after primer application remarkably decreased bond strength when the contamination was not rinsed away. If the blood is not rinsed, the adhesion is inhibited severely, regardless of the step at which contamination occurs (Xie & others, 1993; Powers, Finger & Xie, 1995).

In clinical practice there is a relatively high risk of blood contamination on the adherent dentin surface, especially on the dentin at gingival areas like crown margins prior to placement of prostheses. In addition, the BIS-GMA-related monomer included in the resin monomer is known to have hemolytic properties (Fujisawa & others, 1978). This study apparently revealed that blood contamination of the adherent dentin surface did not allow the adhesive resin to achieve its full potential bond strength. Therefore, blood contamination on the adherent dentin surface during placement of prostheses was likely to be one of the main causes of marginal leakage and secondary caries of the abutment tooth. The results of this study showed that the blood contamination of any conditioned surface immediately prior to bonding markedly reduced bond strength. When blood contamination accidentally occurred on the adherent dentin surface at any step of surface treatment, rinsing the blood with an abundant stream of water was essential as a first step, and subsequent primer application restored the bond strength to that of the controls.

Future research should investigate in more detail the reaction of blood on dentinal apatite or collagen. The incompatibility between bovine dentin and human blood may be discussed. We believe that studies on inhibiting factors of adhesion of the adhesive resin to the dentin can effectively contribute to improving the quality of restorative and prosthetic treatments.

CONCLUSIONS

This study investigated how blood contamination onto the adherent surface can influence bond strengths between an adhesive resin cement and bovine dentin. The results obtained may be summarized as follows:

1. If blood contamination occurred before collagen fibers were exposed by either phosphoric acid etching or self-etching primer application, the contamination had almost no influence on bond strength.

2. Blood contamination of the dentin surface where collagen fibers had been exposed decreased the bond strength even if the blood was washed away with a stream of water and air dried. However, when the contaminated collagen fibers were dissolved or when blood contamination occurred after the exposed collagen fibers were dissolved, the bond strength could be maintained by rinsing the blood away and applying the self-etching dentin primer.

3. The bond strength markedly decreased when blood contamination occurred after the self-etching dentin primer application, but was restored by reapplying the primer.

Based on these results, it is suggested that in the clinical setting the effect of blood contamination on either the smear layer or the collagen layer of the dentin surface can be eliminated by removing the layer. If the contamination occurred on the dentin

where the self-etching dentin primer had already been applied, the contaminated surface should be rinsed with water, air dried, and recoated with the dentin primer to restore the bond strength potential.

(Received 20 November 1998)

References

Ehudin DZ & Thompson VP (1994) Tensile bond strength of dental adhesives bonded to simulated caries-exposed dentin *Journal of Dentistry* **71(2)** 165-173.

Fujisawa S, Imai Y, Kojima K & Masuhara E (1978) Studies on hemolytic activity of bisphenol A diglycidyl methacrylate (BIS-GMA) *Journal of Dental Research* **57(2)** 98-102.

Hansson O (1994) Clinical results with resin-bonded prostheses and an adhesive cement *Quintessence International* **25(2)** 125-132.

Hussey DL, Pagni C & Linden GJ (1991) Performance of 400 adhesive bridges fitted in a restorative dentistry department *Journal of Dentistry* **19(4)** 221-225.

Inagaki A, Chigira H, Itoh K & Wakumoto S (1989) Effects of self-etching primers on dentin *Dental Materials* **5(6)** 403-407.

Isidor F & Stokholm R (1992) Resin-bonded prostheses for posterior teeth *Journal of Prosthetic Dentistry* **68(2)** 239-243.

Johnson ME, Burgess JO, Hermesch CB & Buikema DJ (1994) Saliva contamination of dentin bonding agents *Operative Dentistry* **19(6)** 205-210.

Krejci I, Lutz F & Perisic U (1992) The effects of the processing technic on dentinal adhesion *Schweizerische Monatsschrift Zahnmedizin* **102(8)** 924-929.

Nakabayashi N, Ashizawa M & Nakamura M (1992) Identification of a resin-dentin hybrid layer in vital human dentin created *in vivo*: durable bonding to vital dentin *Quintessence International* **23(2)** 135-141.

Nakabayashi N & Takarada K (1992) Effect of HEMA on bonding to dentin *Dental Materials* **8(2)** 125-130.

O'Brien JA, Retief DH, Bradley EL & Denys FR (1987) Effects of saliva contamination and phosphoric acid composition on bond strength *Dental Materials* **3(6)** 296-302.

Paszyna C, Kerschbaum T, Marinello CP & Pfeiffer P (1990) Clinical long-term results with bonded bridges *Deutsche Zahnarztliche Zeitschrift* **45(7)** 406-409.

Plasmans PJ, Creugers NH, Hermsen RJ & Vrijhoef MMA (1996) The influence of absolute humidity on shear bond adhesion *Journal of Dentistry* **24** 425-428.

Powers JM, Finger WJ & Xie J (1995) Bonding of composite resin to contaminated human enamel and dentin *Journal of Prosthodontics* **4** 28-32.

Sheen DH, Wang WN & Tarng TH (1993) Bond strength of younger and older permanent teeth with various etching times *Angle Orthodontist* **63(3)** 225-230.

Suzuki K & Nakai H (1993) Adhesion of restorative resin to tooth substance–treatment of acid-etched dentin by aqueous solution of HEMA *Journal of the Dental Materials and Devices* **12** 34-44.

Terada R (1993) Characterization of enamel and dentin surfaces after removal of temporary cement–study on removal of temporary cement *Dental Materials Journal* **12** 18-28.

Tjan AH & Nemetz H (1992) Effect of eugenol-containing endodontic sealer on retention of prefabricated posts luted with an adhesive composite resin cement *Quintessence International* **23(12)** 839-844.

Wakabayashi Y, Kondou Y, Suzuki K, Yatani H & Yamashita A (1994) Effect of dissolution of collagen on adhesion to dentin *International Journal of Prosthodontics* **7(4)** 302-306.

Wang T & Nakabayashi N (1991) Effect of 2-(methacryloxy) ethyl phenyl hydrogen phosphate on adhesion to dentin *Journal of Dental Research* **70(1)** 59-66.

Watanabe EK, Yamashita A, Imai M, Yatani H & Suzuki K (1997) Temporary cement remnants as an adhesion inhibiting factor in the interface between resin cements and bovine dentin *International Journal of Prosthodontics* **10(5)** 440-452.

Williams VD, Thayer KE, Denehy GE & Boyer DB (1989) Cast metal resin-bonded prostheses: A 10-year retrospective study *Journal of Prosthetic Dentistry* **61(4)** 436-441.

Xie J, Powers JM & McGuckin RS (1993) *In vitro* bond strength of two adhesives to enamel and dentin under normal and contaminated conditions *Dental Materials* **9(5)** 295-299.

Yamashita A & Tsuji K (1997) Epidemiological evaluation of the clinical results obtained with the adhesion technique *The Journal of the Japan Prosthodontic Society* **41** 895-901.

©Operative Dentistry, 2000, **25,** 202-208

Effect of Food-Simulating Liquids on the Flexual Strength of Composite and Polyacid-Modified Composite Restoratives

AUJ Yap • DTT Tan • BKC Goh
HG Kuah • M Goh

Clinical Relevance

The flexural strengths of the composites were, in general, significantly higher than their polyacid-modified counterparts after conditioning in various aqueous solutions. Clinical usage of polyacid-modified composites in stress-bearing areas should, therefore, be done with caution.

SUMMARY

This study investigates the effects of food-simulating liquids on composite and polyacid-modified composite restoratives. Three composite (Z100, Spectrum TPH, and Tetric Ceram) and three polyacid-modified composite (F2000, Dyract AP, and Compoglass) restoratives from the same manufacturers were selected for the study. Flexural strength specimens (25 x 2 x 2 mm) based on ISO 4049 specifications were fabricated according to the manufacturers' recommendations. After light polymerization, the specimens were removed from their molds and conditioned for one week at 37°C in the following mediums: (1) deionized water, (2) 0.02 M citric acid, (3) heptane, and (4) 50% ethanol-water solution. Specimens stored in air were used as controls. The sample size was five for each material-medium combination. After conditioning, the specimens were blotted dry, measured, and subjected to flexural strength testing using an Instron Universal Testing Machine with a crosshead speed of 0.05 mm/minute. With the exception of Compoglass, flexural strength of all restoratives after conditioning in heptane was significantly greater than that after conditioning in all other mediums and the control. Although no significant difference in flexural strength values was observed between the different restoratives when the materials were conditioned in heptane or air (control), significant differences were observed between the different restoratives after conditioning in aqueous solutions (water, citric acid, and ethanol-water solution). The flexural strengths of the composites were generally significantly higher than their polyacid-modified counterparts after conditioning in the various aqueous solutions. The detrimental effects of aqueous solutions on flexural strength appeared to be greater with polyacid-modified composite resins than with composite restoratives.

National University of Singapore, 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore

Adrian UJ Yap, BDS, MSc, FRSH, assistant professor, Department of Restorative Dentistry, Faculty of Dentistry. Assistant director, Center for Biomedical Materials Applications and Technology

Derek TT Tan, student

Benny KC Goh, student

HG Kuah, student

Monique Goh, student

INTRODUCTION

The search for the ideal restorative material to replace tooth tissue and the demand for products with adhesive and caries-protective properties together with a simple

clinical application procedure have led to the development of restorative materials that combine conventional glass ionomers and light-cure composite resins. Two types of hybrid restoratives, which more or less resemble one of the two basic materials, are currently available. The first is resin-modified glass-ionomer cements, which consist essentially of two components, a powder of fluoroaluminosilicate glass, and a liquid of water-soluble polyacrylic acid with pendant light-sensitive side-chains that can cross-link and thus polymerize. Polymerization can be light- and/or chemically initiated (Sidhu & Watson, 1995). The second is polyacid-modified composite resins, which contain either or both of the essential components of a glass ionomer. The components, however, do not react as part of the setting process (McLean, Nicholson & Wilson, 1994).

Composite resins, as well as glass ionomers, are susceptible to various modes of chemical degradation *in vitro* (Wu & McKinney, 1982; Asmussen, 1984; McKinney & Wu, 1985; McKinney, Antonucci & Rupp, 1987). The mechanical properties of various composites had been tested after long-term storage in solutions. The effects of such aging appear to be dependent upon composition, as well as testing mode. Reductions in fracture toughness after storage for up to one year in water and aqueous solutions containing salt or sucrose have been reported, the magnitude being dependent upon filler composition (Lloyd, 1984). A significant decrease in creep and decrease in flexural strength had also been demonstrated for certain composites after three months of storage in water (Øysaed & Ruyter, 1986). McKinney and Wu (1985) have demonstrated reduced hardness and wear resistance for composites stored for one week in several aqueous solutions of ethanol with different solubility parameters.

In an *in vivo* situation, it can be assumed that saliva, food components, and beverages can degrade and age dental restoratives. Wu and others (1984) reported that clinically damaged composite restorations had altered layers on both nonstress-bearing and on stress-bearing occlusal surfaces. It was speculated that chemical or thermal environment contributed to the *in vivo* degradation of these materials. Thus interactions among the many substances in the oral cavity at 37°C probably have a negative impact on the durability of dental restorations. Although the effects of food-simulating liquids on composites had been extensively studied, few, if any, had been conducted on polyacid-modified composite restoratives.

There is a limit to the value of applied force to which a restoration can withstand without fracturing. The stress at fracture is normally used to characterize the strength of the material. As some polyacid-modified composites are now indicated for stress-bearing areas, such as posterior and Class IV restorations, the knowledge of how their strength properties are affected by food-simulating liquids is important for predicting clinical performance. The objective of this study was to investigate the effects of food-simulating liquids on the flexural strength of composite and polyacid-modified composite resins. Intermaterial comparison after conditioning in the various mediums was also performed.

METHODS AND MATERIALS

Three composite (Z100, Spectrum TPH, and Tetric Ceram) and three polyacid-modified composite (F2000, Dyract AP, and Compoglass F) restoratives from the same manufacturers were selected for the study. The technical profiles of the restoratives and their manufacturers are shown in Table 1.

Flexural-strength testing specimens of the various restoratives were fabricated according to ISO 4049 specifications (25 mm length x 2 mm width x 2 mm height) in customized stainless-steel molds. The restoratives were placed into the mold, which was positioned on top of a glass slide. A second glass slide was then placed on top of the mold, and gentle pressure was applied to extrude excess material from the mold. The top and bottom surfaces of the specimens were then light polymerized in three overlapping irradiations of 40 seconds each with the Max polymerization unit (Dentsply/Caulk, Milford, DE 19963). Immediately after light polymerization, the flash was removed, and the test specimens were separated from their molds. Twenty-five specimens were made for each material. These were randomly divided into five groups of five and conditioned for one week at 37°C as follows: air (control), distilled water, 0.02M citric acid, heptane, 50% ethanol-water solution.

At the end of the conditioning period, the flexural strength of the restoratives was assessed. The specimens were first blotted dry with filter paper, sized with sandpaper, and measured using digital veneer calipers (Mitutoyo Corp, Tokyo, Japan). Measurements were taken in two locations for length, width, and height, and the average of the two values was taken to calculate the flexural strength. Flexural strength testing (Figure 1) was done with an Instron Universal Testing Machine (model 4502; Instron Corp, Canton, MA 02021) at a crosshead speed of 0.05 mm/minute until the specimens fractured. The specimens were aligned to allow for centering of the load. The maximum load exerted on the specimens was recorded, and flexural strength was calculated as s, in megapascals (MPa), using the following equation: $s = 3\,FL/(2\,BH^2)$; where F is the maximum load, in newtons, exerted on the specimens; L is the distance, in millimeters, between the supports, accurate to ± 0.01 mm; B is the width, in millimeters, of the specimen measured immediately prior to testing; H is the height, in millimeters, of the specimens measured immediately prior to testing.

Table 1: *Technical profiles and manufacturers of the materials evaluated.*

Material	Manufacturer	Type	Resin	Filler	Filler size (μm)	Filler content % by weight	Lot No.
Z100	3M Dental Products St Paul, MN 55144	Universal	BisGMA TEGDMA	Zirconia Silica	0.04-3.5	84.5	19980203
Spectrum TPH	Dentsply DeTrey Konstanz, Germany	Universal	BisGMA-adduct BisEMA TEGDMA	Bariumaluminum -borosilicate, Silica	0.04-5	77	970900978
Tetric Ceram	Vivadent, Schaan Liechtenstein	Universal	BisGMA UDMA TEGDMA	Barium glass, Bariumalumino-flurosilicate glass, Mixed oxide, Silica, Ytterbium trifluoride	0.04-1.0	78.6	19972623
F2000	3M Dental Products St Paul, MN 55144	Anterior	CMDA GDMA	Fluroalumino-silicate glass, Silica	3-10	84	19970929
Dyract AP	Dentsply DeTrey Konstanz, Germany	Universal	UDMA TCB	Strontium-fluro-silicate glass	0.8 (mean)	73	9801001150
Compoglass F	Vivadent, Schaan Liechtenstein	Anterior	UDMA PEGDMA DCDMA	Bariumalumino-flurosilicate glass, Ytterbium trifluoride	1.0 (mean)	71.1	909418

BisEMA = Ethoxylated bisphenol-A-glycidyl methacrylate
BisGMA = Bisphenol-A-glycidyl methacrylate
BisGMA-adduct = Adduct of 2,2-Bis[4-2-hydroxy-3-methacryloyloxpropoxy)-phenyl]propane with hexamethylene diisocyanate
CDMA = Dimethacrylate functional oligomer derived from citric acid
DCDMA = Cycloaliphatic dicarbonic acid dimethacrylate
GDMA = Glyceryl methacrylate
PEGDMA = Polyethylene glycoldimethacrylate
TEDGMA = Triethylene glycol dimethacrylate
TCB = Reaction product butane tetracarboxylic acid and HEMA
UMDA = Urethane dimethacrylate

The data were subjected to statistical analysis using one-way ANOVA and post hoc Scheffé's test at a significance level of 0.05. The flexural strength of individual materials after conditioning in the different mediums was compared to study the effects of food-simulating liquids on flexural strength. Intermaterial comparison was also done based on conditioning mediums.

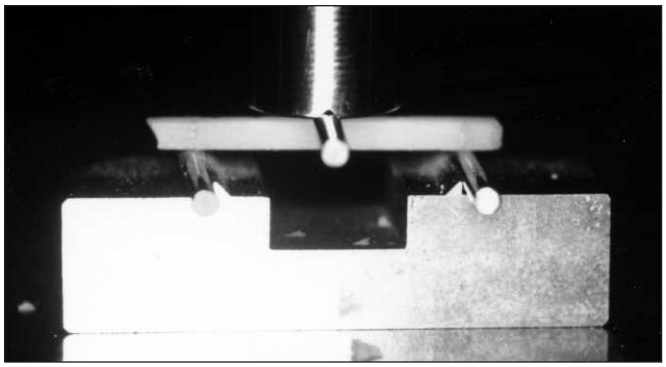


Figure 1. *The flexural strength testing set-up which gives a three-point loading to the specimen.*

RESULTS

The mean flexural strength values ($x10^{-3}$ MPa) of the restoratives after conditioning is reflected in Table 2. Figures 2 and 3 show the mean flexural strength of composites and polyacid-modified composites, respectively. The results of the statistical analysis based on materials are shown in Table 3, and those based on conditioning mediums are shown in Table 4. For all composite and polyacid-modified composite restoratives, the highest flexural strength was obtained after conditioning in heptane. The flexural strength of the air-control specimens ranked second. With the exception of F2000, conditioning in ethanol-water solution resulted in the lowest flexural strength.

For Compoglass (Table 3), flexural strength values after conditioning in heptane were significantly

Table 2: *Mean flexural strength values (x10³ MPa) with standard deviations*

Material	Air (Control)	Water	Citric Acid	Heptane	Ethanol-water solution
Z100	126 (9.81)	90.6 (0.33)	102 (6.58)	251 (2.27)	89.5 (4.26)
Spectrum	142 (8.36)	138 (0.05)	125 (9.30)	264 (9.38)	116 (4.90)
Tetric Ceram	127 (3.67)	115 (4.46)	107 (0.48)	242 (4.73)	60.4 (0.38)
F2000	124 (0.486)	54.8 (0.35)	58.0 (0.67)	265 (6.76)	68.4 (0.96)
Dyract AP	123 (3.64)	82.3 (3.42)	89.0 (3.57)	197 (8.85)	36.5 (0.16)
Compoglass F	128 (3.18)	68.1 (2.48)	91.8 (0.06)	186 (4.56)	17.1 (0.23)

Table 3: *Results of statistical analysis based on materials*

Material	Differences
Z100	Heptane > Air (control), Water, Citric Acid, Ethanol-water solution
Spectrum	Heptane > Air (control), Water, Citric Acid, Ethanol-water solution
Tetric Ceram	Heptane > Air (control), Water, Citric Acid, Ethanol-water solution
F2000	Heptane > Air (control), Water, Citric Acid, Ethanol-water solution
Dyract AP	Heptane > Air (control), Water, Citric Acid, Ethanol-water solution
Compoglass F	Heptane > Air (control), Water, Citric Acid, Ethanol-water solution Air (control), Citric Acid > Ethanol-water solution

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

Table 4: *Results of statistical analysis based on conditioning mediums*

Conditioning Mediums	Differences
Air (Control)	No significant difference
Distilled Water	Spectrum, Tetric > Z100, F2000, Dyract, Compoglass Z100, Dyract > F2000
Citric acid	Z100, Spectrum, Tetric > F2000 Spectrum > Dyract
Heptane	No significant difference
50% Ethanol-water Solution	Spectrum > Z100, Tetric, F2000, Dyract, Compoglass Z100 > Tetric, Dyract, Compoglass Tetric > Dyract, Compoglass

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

greater than those after conditioning in water, citric acid, and ethanol-water solution. No significant difference in flexural strength values was observed between specimens conditioned in heptane and the control (air). Flexural strength values for specimens conditioned in ethanol-water solution were significantly lower than that obtained for the control and specimens conditioned in citric acid.

For all other restoratives evaluated (ie, Z100, Spectrum, Tetric, F2000, and Dyract), flexural strength values obtained after conditioning in heptane were significantly greater than those after conditioning in all other mediums and the control (Table 3). No significant difference in flexural strength values was observed between the control and specimens conditioned in water, citric acid, and ethanol-water solution.

No significant difference in flexural strength values was observed between the different restoratives when the materials were conditioned in heptane or air (control). When conditioned in water, Spectrum and Tetric had significantly greater flexural strength than Z100 and all the polyacid-modified composites (Table 4). The flexural strength of Z100 and Dyract in water was significantly greater than that of F2000. When conditioned in citric acid (Table 4), all the composite restoratives had significantly higher flexural strength than F2000. Spectrum was also significantly higher than Dyract. After conditioning in ethanol-water solution (Table 4), the flexural strength of Spectrum was significantly higher than that of all the other restoratives. Z100 was significantly greater than Tetric, Dyract, or Compoglass, and Tetric was greater than that of Dyract and Compoglass.

Figure 2. Mean flexural strength of composite resins.

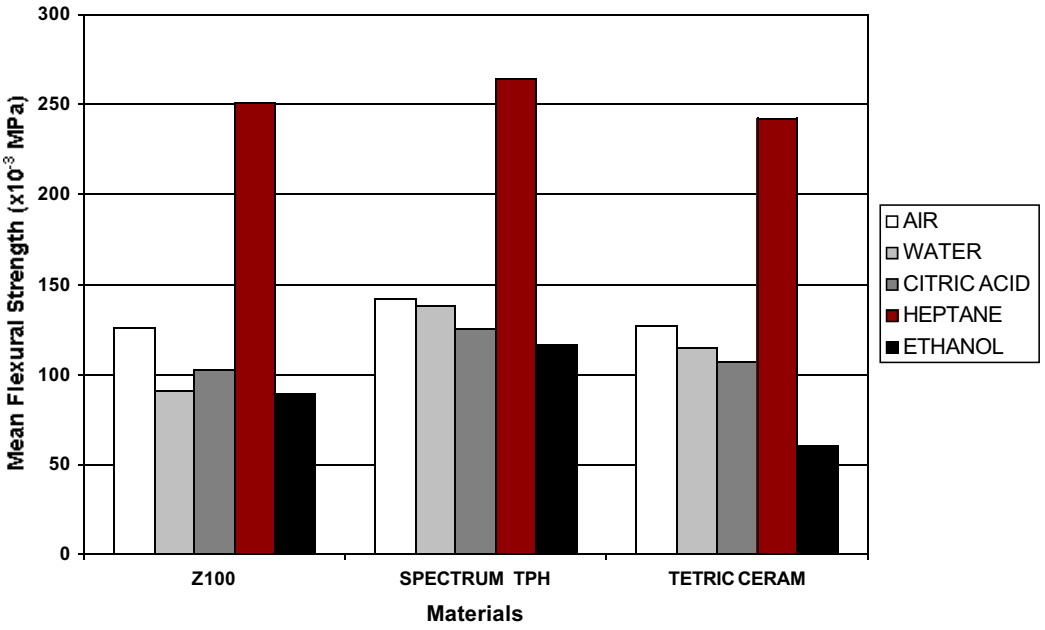
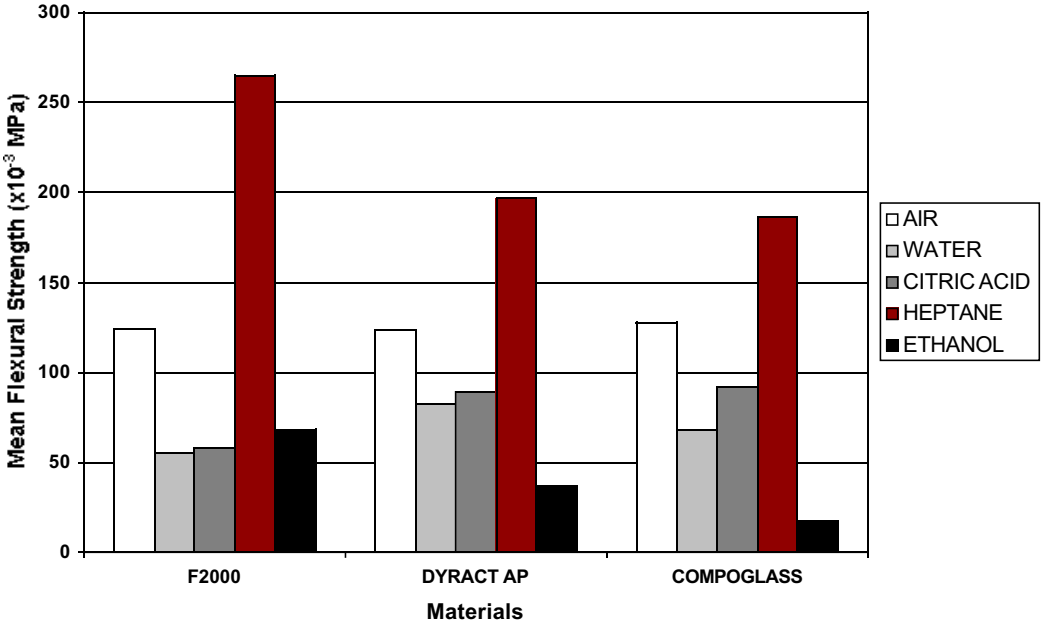


Figure 3. Mean flexural strength of polyacid-modified composite resins.



DISCUSSION

The food-simulating liquids used for conditioning the restoratives in this investigation are among those recommended in FDA Guidelines (FDA, 1976) to be used as food simulators. Heptane simulates, for example, butter, fatty meats, and vegetable oils. The ethanol solution and citric acid simulate certain beverages, including alcoholic, and vegetables, fruits, candy, and syrup. Deionized water was included to simulate the wet intraoral environment provided by saliva and water. The period of conditioning before flexural strength testing was one week without interruption. This length of time was rather extensive in the light of the fact that restoratives come into contact with foods only briefly in a sporadic way (during eating and drinking until teeth are cleaned). Accordingly, the test results reported might exaggerate the effects that chemicals have on the flexural strength of the restoratives. Continuous exposure may, however, occur *in vivo*, as chemical agents can be absorbed by adherent debris (such as tartar or food particles) at the margins of restorations, or they can be produced by the decomposition of debris. The reported increase or decrease in flexural strength values measured herein may exceed that which occurs *in vivo*. The accelerated *in vitro* rate, however, provides an important clue to the performance of these composites under stress-bearing conditions *in vivo*.

For all restoratives evaluated, a significant increase in flexural strength as compared to conditioning in other aqueous mediums was observed after conditioning in heptane. With the exception of Compoglass, con-

ditioning in heptane also resulted in significantly higher flexural strength values than the control. Two possible explanations exist for this observation. First, heptane reduces oxygen inhibition (Ruyter, 1981) during post-curing that occurs for specimens conditioned in air (control). Second, heptane eliminates leaching out of silica and combined metals in fillers, which may occur from conditioning in aqueous solutions (Söderholm, 1983). The latter also accounts for the higher flexural strengths of control specimens compared to those conditioned in aqueous mediums. All restoratives evaluated contained either silica or silicate glass fillers, which have irregularly distributed Si-O-Si bonds. When the different resin-bonded fillers are immersed in water, the resin matrices will swell, and radial tensile stresses will be introduced at the filler interfaces, thereby straining the Si-O-Si bonds in the fillers. The high energy levels resulting from strained Si-O-Si bonds make the fillers more susceptible to stress corrosion attacks (Söderholm, 1983). The attack of water on the stressed Si-O-Si bonds can be written as: $Si_2O + H_2O = 2$ SiOH. Complete or partial filler debonding occurs due to stress corrosion at the surface layer of fillers. This degradation of the filler-matrix interface results in decreased flexural strengths and other physical properties (Söderholm, 1982; Söderholm & Roberts, 1990).

Hoop stresses also exist around the filler particles as a result of matrix shrinkage during polymerization (Söderholm, 1984). These hoop stresses increase the frictional forces between the filler and resin matrix, thereby decreasing the filler pull-out tendency during flexural strength testing. However, when the restoratives are exposed to aqueous solutions, the plasticizing and swelling of the matrix reduces the hoop stresses around the fillers, which will facilitate filler pull-out, thus decreasing flexural strength. A protective layer over the surface of composites may therefore enhance the physical properties of composites and polyacid-modified composites.

With the exception of F2000, conditioning in ethanol-water solution resulted in the lowest flexural strength. The resin matrices of Z100, Spectrum, Tetric, Dyract, and Compoglass are based either on BIS-GMA or UDMA. A close match in solubility parameters results in maximum softening of the resin (Wu & McKinney, 1982; McKinney & Wu, 1985). As the solubility parameter values for 75% to 50% aqueous ethanol solution (3 to 3.7 x 10^4 $J^{1/2}$ $m^{-3/2}$) approximates that of BIS-GMA and UDMA, softening effects on composites and polyacid-modified composites based on these oligomers are expected to be greatest (Kao, 1989). The extent of damage may depend somewhat on the diffusion rate, which, in turn, depends on the molecular weight of the penetrant. For the most part, the damage mechanism had been attributed to the softening of the polymer matrix, which results in its partial removal at the surface (McKinney & Wu, 1985). The partial removal of the resin matrix results in the "prouding" of the filler particles, which serve as areas of stress concentration during flexural strength testing. This may, in part, account for the lower flexural strength noted after conditioning in ethanol-water solution.

F2000 is based on CMDA, which is a dimethacrylate functional oligomer derived from citric acid. The CDMA is similar in composition and function to the methacrylated polycarboxylic acid copolymer used in Vitrebond liner/base, Vitremer restorative, and some other 3M adhesive products. GDMA, the other resin component of F2000, is chemically and functionally similar to HEMA (hydroxyethyl methacrylate), and like HEMA, it has hydroxyl functionality, which makes it hydrophilic. GDMA acts as a diluent for CDMA, and copolymerizes with the oligomer. It is conceivable that the solubility parameter of CDMA is closer to that of citric acid, which it has been derived from, than a 50% ethanol-water solution. The possible removal of the CDMA resin matrix and the absorption of water by GDMA may account for the lower flexural strength observed with F2000 after conditioning in citric acid and deionized water.

The effects of food-simulating liquids may only be a surface phenomenon. The surface of materials is usually affected before the bulk (Braden, Causton & Clarke, 1976). This explains the general lack of statistical significance in flexural strength values between control specimens and specimens conditioned in water, citric acid, and ethanol-water solution, despite the lower flexural strength values observed after one week of conditioning. A longer conditioning period may, however, result in greater statistical significance. For Compoglass, control specimens and specimens conditioned in citric acid had significantly higher flexural values than specimens conditioned in ethanol-water solution. Among the different restoratives, the filler content of Compoglass was the lowest. This means that for every standard surface area, a greater area of resin will be exposed to the conditioning mediums. This, in addition to the fact that maximum softening of urethane dimethacrylate matrix (which Compoglass is based upon) results from exposure to 50% ethanol-water solution (Kao, 1989), contributes to the significant difference between the control and specimens conditioned in ethanol-water solution.

When the materials were compared based on mediums, no significant difference in flexural strength values was observed between the different restoratives when they were conditioned in heptane or air (control). When conditioned in aqueous solutions (ie, water, citric acid, and ethanol-water solution), significant difference in flexural strength values was noted between the

different restoratives. The flexural strengths of the composites were generally significantly higher than their polyacid-modified counterparts after conditioning in the various aqueous solutions. The detrimental effects of aqueous solutions on flexural strength thus appear to be greater with polyacid-modified composite resins. Clinical usage of polyacid-modified composites in stress-bearing areas should therefore be done with caution. The lower flexural strength values of polyacid-modified composites may be attributed, in part, to the uptake of water, which is necessary for the activation of the acid-base reaction within the polymer matrix (Dentsply, 1994).

For the materials evaluated, Spectrum consistently gave the highest flexural strength after conditioning in water, citric acid, and ethanol-water solution. Spectrum utilizes BIS-EMA and modified BIS-GMA. The significantly higher flexural strength noted may be contributed in part by the hydrophobic nature of the ethoxylated version of BIS-GMA (ie, BIS-EMA), which does not contain any unreacted hydroxyl groups on the main polymer chain (Ruyter & Nilsen, 1993).

CONCLUSIONS

The effect of food-simulating liquids on the flexural strength of composites and polyacid-modified composites was investigated. For all composite and polyacid-modified composite restoratives, the highest flexural strength was obtained after conditioning in heptane. The flexural strength of all restoratives after conditioning in heptane was generally significantly greater than that after conditioning in all other mediums and the control. With the exception of F2000, conditioning in ethanol-water solution resulted in the lowest flexural strength.

No significant difference in flexural strength values was observed between the different restoratives when the materials were conditioned in heptane or air (control). Significant differences in flexural strengths were, however, observed between the different restoratives after conditioning in aqueous solutions (ie, water, citric acid, and ethanol-water solution). The flexural strengths of the composites were generally significantly higher than their polyacid-modified counterparts after conditioning in aqueous solutions. The detrimental effects of aqueous solutions on flexural strength appeared to be greater with polyacid-modified composite resins than composite restoratives.

Acknowledgements

The authors would like to thank Dr Ming Li for her assistance and professional advice.

(Received 10 December 1998)

References

Amussen E (1984) Softening of BIS-GMA-based polymers by ethanol and by organic acids of plaque *Scandinavian Journal of Dental Research* **92** 257-261.

Braden M, Causton EE & Clarke RL (1976) Diffusion of water in composite filling materials *Journal of Dental Research* **55(5)**730-732.

Dentsply (1994) Dyract–A Single Component Compomer. The Technology Behind It Weybridge, England: *DeTrey-Dentsply International* pp 5-9.

Food and Drug Administration (1976) FDA Guidelines for Chemistry and Technology Requirements of Indirect Additive Petitions Washington, DC: FDA.

Kao EC (1989) Influence of food-simulating solvents on resin composites and glass-ionomer restorative cement *Dental Materials* **5** 201-208.

Lloyd CH (1984) The fracture toughness of dental composites III. The effect of environment upon the stress intensification factor (KIC) after extended storage *Journal of Oral Rehabilitation* **11** 393-398.

McKinney JE & Wu W (1985) Chemical softening and wear of dental composites *Journal of Dental Research* **64(11)** 1326-1331.

McKinney JE, Antonucci JM & Rupp NW (1987) Wear and microhardness of glass-ionomer cements *Journal of Dental Research* **66(6)** 1134-1139.

McLean JW, Nicholson JW & Wilson AD (1994) Proposed nomenclature for glass-ionomer dental cements and related materials *Quintessence International* **25(9)** 587-589.

Øysaed H & Ruyter IE (1986) Composites for use in posterior teeth: mechanical properties tested under dry and wet conditions *Journal of Biomedical Materials Research* **20(2)** 261-271.

Ruyter IE (1981) Unpolymerized surface layers on sealants *Acta Odontologica Scandinavica* **39** 27-32.

Ruyter IE & Nilsen J (1993) Chemical characterization of six posterior composites *Journal of Dental Research* **72** Abstracts of Papers p 177 Abstract 588.

Sidhu SK & Watson TF (1995) Resin-modified glass ionomer materials. A status report for the *American Journal of Dentistry* **8(8)** 59-67.

Söderholm K-JM (1982) Relationship between compressive yield strength and filler fractions of PMMA composites *Acta Odontologica Scandinavica* **40** 145-150.

Söderholm K-JM (1983) Leaking of fillers in dental composites *Journal of Dental Research* **62(2)** 126-130.

Söderholm K-JM (1984) Influence of silane treatment and filler fraction on thermal expansion of composite resins *Journal of Dental Research* **63(11)** 1321-1326.

Söderholm K-JM & Roberts MJ (1990) Influence of water exposure on the tensile strength of composites *Journal of Dental Research* **69(12)** 1812-1816.

Wu W & McKinney JE (1982) Influence of chemicals on wear of dental composites *Journal of Dental Research* **61(10)** 1180-1183.

Wu W, Toth EE, Moffa JF & Ellison JA (1984) Subsurface damage layer of *in vivo* worn dental composite restorations *Journal of Dental Research* **63(5)** 675-680.

©Operative Dentistry, 2000, **25,** 209-215

Repair Strength of Etched vs Silica-Coated Metal-Ceramic and All-Ceramic Restorations

R Frankenberger • N Krämer • J Sindel

Clinical Relevance

Silica coating represents an effective method for repair of porcelain fused-to-metal and all-ceramic restorations, enabling simultaneous treatment of ceramic and metal.

SUMMARY

The purpose of this *in vitro* study was to examine shear bond strengths of composite resin to metal-exposed porcelain-fused-to-metal (PFM) and all-ceramic restorations after silica coating or etching with 5% hydrofluoric acid (HF).

Specimens were fabricated for each of the following groups: two all-ceramic materials [a feldspathic porcelain (Vita Mark II) and a leucite-reinforced glass-ceramic (IPS Empress)], and one noble metal-ceramic (Orplid Keramik I alloy; Vita VMK 68 N feldspathic veneer ceramic). These groups were repaired with resin composites after different pretreatment methods. In one metal-ceramic subgroup the surface exhibited a 50% metal and 50% ceramic exposure. In the silica-coating groups, the specimen surfaces were air abraded with silica acid-modified Al_2O_3 (CoJet Sand) and treated corresponding to the porcelain repair with resin composite. For control groups, the surfaces were etched with 5% HF for 60 seconds and treated in the same way as the silica-coated groups. After 24 hours of storage (distilled water, 37°C) and an additional 24 hours of thermocycling (1150 x 5°C/55°C) the specimens were debonded using a shear bond strength test (n=15).

In all groups the silica coat repair achieved equal or significantly higher bond strengths than did the etching technique ($p<0.05$, Mann-Whitney U test). In the metal-exposed group, the mean bond strength increased from 7.3 MPa to 16.3 MPa following the silica-coat repair. Results indicated that silica coating represents a suitable treatment for the intraoral repair of the materials tested in the present study.

Policlinic for Operative Dentistry and Periodontology, University of Erlangen-Nuremberg, Erlangen, Germany

Roland Frankenberger, DMD, assistant professor

Norbert Krämer, DMD, PhD, associate professor

Jürgen Sindel, Dipl Eng, research associate

INTRODUCTION

Due to their high esthetic qualities and their mechanical stability in the oral environment, porcelain-fused-to-metal (PFM) restorations are commonly used in daily dental practice (Kelly, Nishimura & Campbell, 1996). However, clinical studies show failure rates up to 9% for ceramic veneers (Bronnimann, Fritzsche & Schärer, 1991).

Complete removal of the fractured restoration is unpleasant and expensive for the patient; therefore,

the possibility to repair failed PFM or all-ceramic restorations intraorally is a clinical challenge (Rada, 1991; Appeldoorn, Wilwerding & Barkmeier, 1993; Kupiec & others, 1996; van der Vyver, Marais & de Wet, 1996).

Success of intraoral repair has been a problem when metal is exposed (Schneider, Powers & Pierpont, 1992; Zhukovsky & others, 1996). The intraoral ceramic fracture repairs using different repair systems have achieved promising bond strengths after etching with hydrofluoric acid (HF) or using air abrasion as pretreatment methods (Della Bona & van Noort, 1995; Chung & Hwang, 1997; Chen, Matsumura & Atsuta, 1998).

For repair of multi-unit fixed partial dentures, different methods have been suggested, such as resin bonded overcastings, but compared with composite-to-ceramic repair, these types of treatment are more time-consuming (Matsumura & Atsuta, 1996).

Veneering of metal alloys with composite resins using silica coating is a well-established method used in dental laboratories with acceptable clinical results (Hummel, Pace & Marker, 1994; Schneider & others, 1992). A new method for intraoral silica coating as modification of the laboratory methodology promises to enhance the clinical performance of composite-repaired metal-exposed PFM restorations. Additionally, the pretreatment of fractured all-ceramic restorations may be possible using this technique.

The purpose of the present *in vitro* study was to examine shear bond strengths of resin composite to simulated all-ceramic and metal-exposed PFM restorations after surface pretreatment with either 5% HF etching or silica coating.

METHODS AND MATERIALS

Shear bond strengths of resin composite cylinders bonded to two ceramic materials and one metal-ceramic surface as a repair simulation were investigated in the present *in vitro* study.

Specimen Preparation

The metal-ceramic samples were produced as follows: The opaque and body veneering porcelain VMK 68 N (Vita Zahnfabrik, D-79713 Bad Säckingen, Germany) were fired to rectangular (10 x 10 x 1 mm) metal specimens (Au-Pt alloy Orplid Keramik I; Hafner, D-75173 Pforzheim, Germany) with a thickness of approximately 1.5 mm (Figure 1). For the metal-exposed groups, a half-metal specimen was welded to a complete metal specimen using a laser (Haas Industrielaser, Schrammberg, Germany). Consecutively, the deeper area was filled by firing the porcelain (Figure 1). To guarantee a plane surface, the specimens were lapped (PM 2; Logitech, Glasgow, Scotland) with 3 µm Al_2O_3.

The feldspathic porcelain specimens (Vita Mark II; Vita Zahnfabrik) were produced by grinding computer-generated rectangular plates (10 x 10 x 1.5 mm) with the CAD/CAM system Cerec 1 (Siemens, D-64625 Bensheim, Germany).

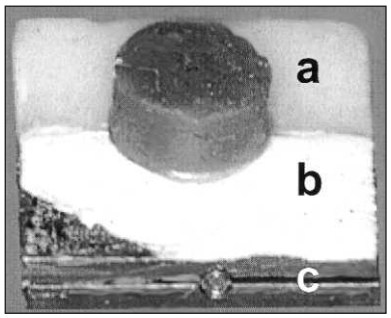


Figure 1. *Composite resin cylinder bonded to metal-exposed ceramic surface: a) veneer ceramic; b) metal with opaquer; c) welded metal plates.*

The leucite-reinforced glass-ceramic specimens (IPS Empress; Ivoclar, FL-9494 Schaan, Liechtenstein) were manufactured using replicas of disks (12 mm in diameter, 1.5 mm thick) and hot-pressing the ceramic specimens according to the manufacturer's instructions.

Pretreatment of Specimens for Repair

To simulate a clinical situation, all samples were finished with a water-cooled diamond bur (54-76 µm, Komet; Brasseler, D-32657 Lemgo, Germany) mounted in an apparatus with a fixed handpiece to maintain a uniform flat specimen shape.

The sample surfaces were treated with either silica coating or, for the control group, etching with 5% HF. Procedures, materials, and codes for the etching-technique are provided in Table 1.

The first step was to etch the surface with 5% HF for 60 seconds. Subsequently, the surface was silanated (Espe Sil; ESPE, D-82229 Seefeld, Germany) and left for five minutes. Next, the bonding resin was applied, air-thinned, and immediately light cured for 20 seconds.

In the metal-exposed group only the ceramic part was treated with 5% HF; the metal surface was exclusively covered with opaquer (Dentacolor, Batch No 528/45; Kulzer, D-61273 Wehrheim, Germany).

The procedures, materials, and codes for the silica-coated groups are listed in Table 2. An air-abrasion unit prototype (ESPE) was connected to the air pressure of the dental unit. Then the specimens were blasted perpendicular to the surface with silica acid-modified Al2O3 (CoJet Sand; ESPE–particle size 30 µm, pressure 30 psi) from a distance of 10 mm for approximately 15 seconds. After coating the surface, the silane coupling agent was immediately applied and left undisturbed for five minutes. Finally, the bonding agent was painted onto the surface, air-thinned, and light cured according to the etching groups. In the metal-exposed group, the coated metal surface was silanated identically and covered with Dentacolor-opaquer representing the bonding agent.

Table 1

Material	Code	Treatment	Batch Number	Manufacturer
IPS Empress	EM	Ceramics-etch Espe Sil Visio Bond Pertac II	1073 060 104 005	Vita, Bad Säckingen, Germany Espe, Seefeld, Germany Espe Espe
Vita Mark II	VM	Ceramics-etch Espe Sil Visio Bond Pertac II	1073 060 104 005	Vita Espe Espe Espe
Vita VMK 68 N	VMK	Ceramics-etch Monobond S Heliobond Estilux Posterior	1073 906891 613764 experimental (blue shade of commercial product	Vita Vivadent, Schaan, Liechtenstein Vivadent Kulzer, Wehrheim, Germany
Vita VMK 68 N (50% metal exposed)	VMK50	Ceramics-etch Monobond S Heliobond Estilux Posterior	1073 906891 613764 experimental	Vita Vivadent Vivadent Kulzer

Table 2

Material	Code	Treatment	Batch Number	Manufacturer
IPS Empress	EM	CoJet-sand Espe Sil Visio Bond Pertac II	002 060 104 005	Espe, Seefeld, Germany Espe Espe Espe
Vita Mark II	VM	CoJet-sand Espe Sil Visio Bond Pertac II	002 060 104 005	Espe Espe Espe Espe
Vita VMK 68 N	VMK	CoJet-sand Monobond S Heliobond Estilux Posterior	002 906891 613764 experimental	Espe Vivadent, Schaan, Liechtenstein Vivadent Kulzer, Wehrheim, Germany
Vita VMK 68 N (50% metal exposed)	VMK50	CoJet-sand Monobond S Heliobond Estilux Posterior	002 906891 613764 experimental	Espe Vivadent Vivadent Kulzer

SEM Evaluation after Pretreatment

Pretreated surfaces of representative samples were examined using a scanning electron microscope (Leitz ISI SR 50; Akashi, Tokyo, Japan) operated at 20 kV.

Repair Simulation

To simulate the repair with resin composite, a metal form was placed onto the pretreated surface, and a cylindrical hole was filled with composite resin by using a plugger. Excess material was removed from the sample surface with a scaler prior to polymerization. Subsequently, the resin composite cylinder was light cured for 40 seconds. Both bonding agent and resin composite were cured with an Elipar II curing light (ESPE Germany). The metal form was removed and light curing was repeated for another 40 seconds. 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.

The diameter of the bonded resin composite cylinders was 4.04 mm and the thickness approximately 2 mm. In the metal-exposed groups the composite resin cylinder was fixed approximately half on ceramic and half on metal. The exact relationship between the different bonding surfaces was determined using a X20 light microscope (Wild M3Z; Heerbrugg, Switzerland)

Table 3

Group (n=15)	VM	EM	VMK	VMK50
Silica Coating	12.7 (2.7)	15.3 (1.6)[a]	15.3 (3.9)[b]	16.3 (2.8)[c]
Etching	15.8 (4.2)	11.2 (2.2)[a]	11.2 (2.5)[b]	7.3 (1.6)[c]

Data with same superscript letters within groups are significantly different (Mann-Whitney U, $p<0.05$).

equipped with a video camera. Images of the samples were digitized and the different bonding surface amounts were measured with an image processing software (Tiffmess 1.9; University of Erlangen, D-91054 Erlangen, Germany).

Storage and SBS Test

All specimens were stored in distilled water for 24 hours at 37°C. 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; the transport time between the water baths was 15 seconds.

The specimens were mounted in a fixture for determination of shear bond strength. A chisel-shaped rod was attached to a compression load cell traveling at a crosshead speed of 0.5 mm/minute. The resin composite cylinders were loaded parallel to the sample surface until fracture occurred using a Universal Testing Machine (Zwicki; Zwick, D-89075 Ulm, Germany). In the metal-exposed groups, the resin composite cylinders were always loaded from the metal side. Shear bond strength was tested using 15 specimens for each group. The fracture modes of the composite cylinders were examined by SEM and divided into adhesive or cohesive failures.

Statistical Analysis

Statistical analysis was performed using SPSS/PC+ for Windows 95 (Vers 7.5; SPSS Inc, Chicago, IL 60611). The SBS values were nonnormally distributed (seven out of eight groups, proved by Kolmogorov-Smirnov test); therefore, the nonparametric Mann-Whitney U test and Kruskal-Wallis H test for pairwise comparisons at the 0.05 level of significance (a) were performed. The levels of significance were adjusted to a *=1-(1- a)1/k (k=number of performed pairwise tests), to assess the influence of the different pretreatment protocols. The distributions of the strength values were represented in boxplots. The lower boundary of the box is the 25th percentile, the upper boundary represents the 75th percentile. The line within the box is the median of the particular group. The mean 50% of the cases are exhibiting values within each box. Cases with values less than 1.5 of box lengths are marked with lines from the end of the box.

RESULTS

Figure 2 displays the boxplots of the shear bond strengths. The mean shear bond strengths and the standard deviations of the control and of the silicoating groups are listed in Table 3.

In Groups EM, VMK, and VMK50 the silica coating pretreatment achieved significantly higher bond strengths than did the etching technique ($p<0.05$).

Silicatization of the surface in the metal-exposed group resulted in an increase of mean bond strength from 7.3 to 16.3 MPa ($p<0.05$).

In the VM groups, no statistically significant difference between silica coating and etching with 5% HF was observed ($p>0.05$).

In the all-ceramic groups, the resin composite cylinders fractured cohesively (100%), chipping off the ceramic surface.

In the VMK50 group the cylinders failed adhesively between opaquer and metal (100%), and cohesively within the ceramic (100%).

Figures 3-6 show scanning electron micrographs of original surfaces and silica-coated surfaces of each material group. After blasting with the CoJet Sand, a fine and microretentive structure was observed in all micrographs. Partially, blasting particles were baked with the surface representing the effect of this particular tribochemical methodology.

DISCUSSION

The selected test design for determining repair of metal-ceramic and all-ceramic restorations was a shear bond test, which represented a well-suited methodology for simulating intraoral shear stress (Della Bona & van Noort, 1995; Schneider & others, 1992). For simulating metal-exposed failure surfaces, 50-50 metal-ceramic specimens were created and always loaded from the metal side as the weaker element within this construction (Rada, 1991, Zhukovsky & others, 1996).

Furthermore, a recently developed device for intraoral repair (CoJet Sand) as a modification of the well-established Rocatec system (ESPE) needed to be tested and compared with conventional etching using hydrofluoric acid.

Air abrasion using the CoJet Sand entails a tribochemical coating with a ceramic bonding agent similar to the Rocatec system. The blasted particles produce a high spot heat, which together with the blasting pres-

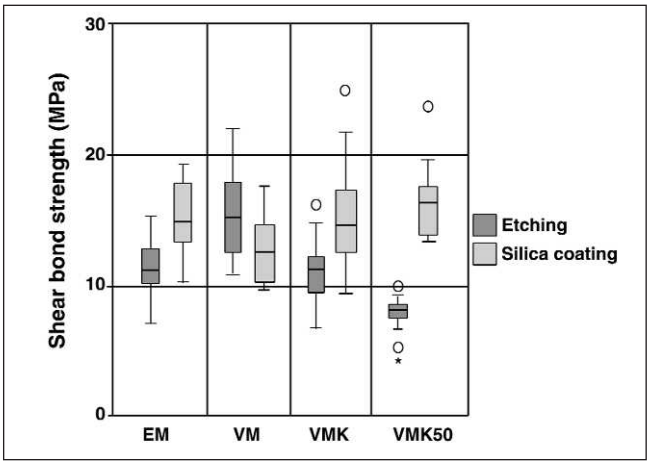


Figure 2. *Box plots of the shear bond strengths.*

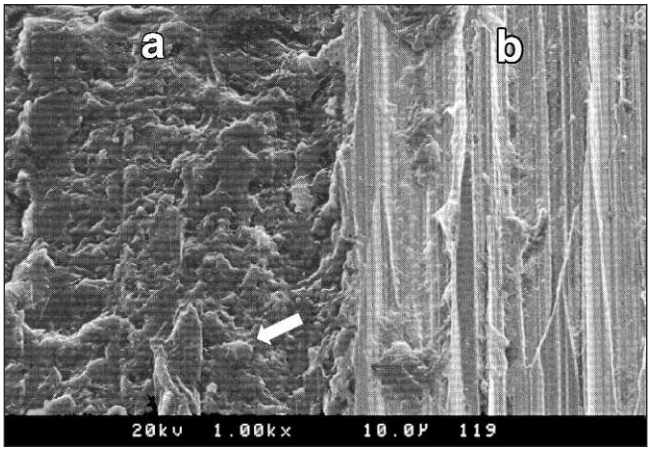


Figure 3. *Scanning electron micrograph of the alloy surface: a) silica-coated, b) finished with diamond bur. The arrow indicates blasting particles baked with the surface.*

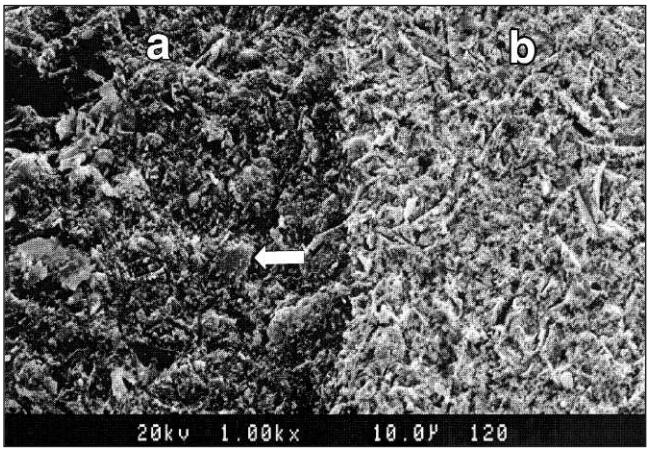


Figure 4. *Scanning electron micrograph of the veneer ceramic surface: a) silica coated, b) finished with diamond bur. The arrow indicates blasting particles baked with the surface.*

sure results in embedding the silica acid-modified aluminum oxide within the material surface (Albers, 1991; Bodenheim & others, 1998). Figures 3-6 clearly demonstrate this process. The two silicatization methods differ in the abrasion parameters. Laboratories use two abrasion steps (110 µm Al_2O_3 and 110 µm silica acid-modified Al_2O_3 at 36 psi), whereas the CoJet Sand (30 µm silica acid-modified Al_2O_3) uses a pressure of 30 psi. With these air-abrasion changes, damage to thin restorations may be avoided.

The high shear bond strengths after silica coating can be explained by two mechanisms that improve the bonding to the repair resin composite: First, as previously reported, the surface roughening resulting from the air-abrasion treatment provides a larger surface area for increased wettability, and simultaneously, a microretentive structure for the micromechanical luting of the bonding material (Phoenix & Shen, 1995). The superiority of the sandblasting procedure over a mechanical roughening with a coarse diamond has been demonstrated (Appeldoorn & others, 1993; Wolf, Powers & O'Keefe, 1992).

The second mechanism is the improved chemical bonding among resin, silane, and silica-coated surfaces, because chemical bonding is a major factor required for good adhesion to metal, whereas micromechanical bonding is less important. For base alloys, different adhesive systems, such as the Liner-M/Clearfil system, have produced high bond strengths and are considered clinically acceptable for intraoral repair of exposed metal (Chung & Hwang, 1997; Wolf & others, 1992). However, when noble alloys were repaired, lower bond strengths resulted, due to the low reactive character of these alloys (Albers, 1991; Ishijima, Caputo & Mito, 1992). This problem can be solved by layering the noble metal with a more reactive material, as shown in the present study exhibiting high bond strengths also within the silica-coated metal-exposed group.

The high bond strengths in the all-ceramic groups after silicatization can be explained by both contributing factors, surface roughening and simultaneously increasing the silica content, which increases the surface area. As a result, the chemical bonding is improved by a higher number of reaction partners for the silane.

This clearly explains the observed significant increase in bond strength of the Groups EM and VMK after silica coating compared to the hydrofluoric acid-etching technique. In the VM group, however, pretreatment with CoJet Sand resulted in a slight, but not statistically significant, decrease in bond strength. Although a different bonding agent/resin composite combination was selected within the Groups VMK and VMK 50 than that in the Groups EM and VM tested with materials of the same manufacturer, these particular VMK groups exhibited favorable SBS results after silica-

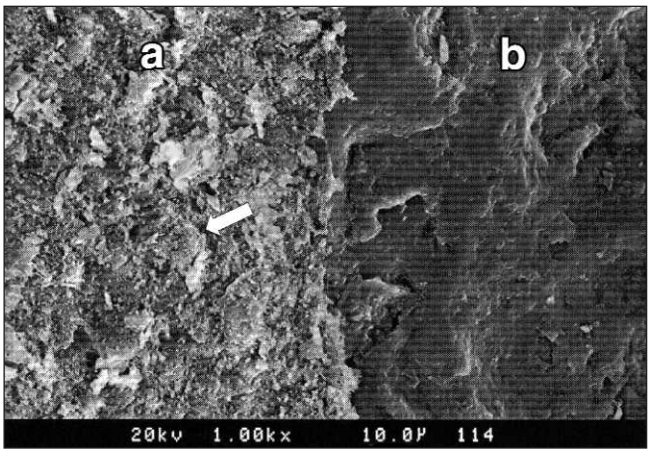


Figure 5. *Scanning electron micrograph of the IPS Empress surface: a) silica coated, b) untreated. The arrow indicates blasting particles baked with the surface.*

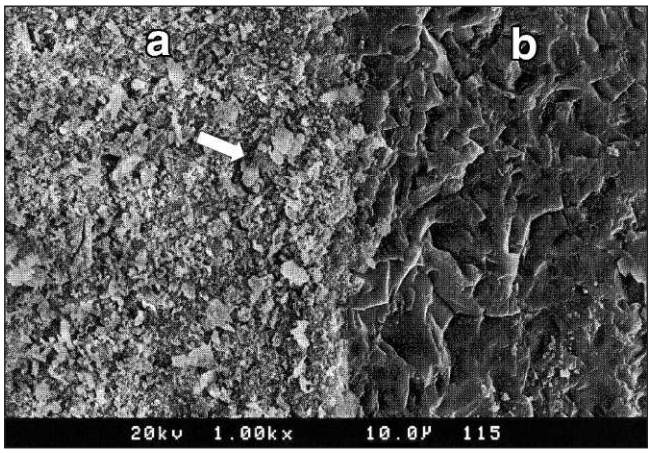


Figure 6. *Scanning electron micrograph of the Vita Mark II surface: a) silica-coated, b) ground with the Cerec 1-system. The arrow indicates blasting particles baked with the surface.*

tization. These results tend to confirm the assumption that the tested silica-coating process is suitable for several brands of repair resin composites.

With respect to the VM results, the efficiency of the silica-coating process seemed to be dependent on the mechanical properties of the air-abraded ceramic. Nevertheless, also in the VM group, silica coating performed as well as using hydrofluoric acid etching, resulting in 100% cohesive fractures as well.

For the particular IPS Empress ceramic, the silica-coating process was reported to be a suitable alternative to hydrofluoric acid etching as inlay pretreatment when luting inlays (Bodenheim & others, 1998). Therefore, based on the present results, silica coating could be recommended as pretreatment for this inlay cementation.

Previous reports have shown that the intraoral repair of fractured porcelain without metal exposure is a solvable problem (Rada, 1991; Appeldoorn & others, 1993). The repair of fracture with both porcelain and metal exposed is more problematic, due to the different characteristics of each particular material. For base alloys, reasonable bond strengths can be obtained using a combination of sandblasting and repair with commercial repair systems; however, repair bond strengths to the corresponding veneering ceramic were often less favorable (Chung & Hwang, 1997). Therefore, completely different pretreatments for both metal and ceramic have to be carried out to obtain maximum bonding of the repair resin composite. In this case, pretreatment with CoJet Sand represented a distinct simplification in repairing metal-exposed failures represented by high bond strengths to both metal and ceramic surfaces, due to the possible simultaneous treatment of the different bonding substrates.

CONCLUSIONS

Within the limits of the present *in vitro* study, it can be concluded:

1. Silica coating with CoJet Sand represents an acceptable alternative to etching with hydrofluoric acid for intraoral repair of the tested metal-exposed porcelain-fused-to-metal and all-ceramic restorations.

2. Especially for metal-exposed restorations, silicatization obtains promising bond strengths.

3. Fractured ceramic and exposed metal surfaces can be pretreated in one step.

(Received 13 December 1998)

References

Albers HF (1991) Metal-resin bonding *Adept Report* **2** 25-40.

Appeldoorn R, Wilwerding TM & Barkmeier WW (1993) Bond strength of composite resin to porcelain with newer generation porcelain repair systems *Journal of Prosthetic Dentistry* **70(1)** 6-11.

Bodenheim G, Sindel J, Frankenberger R, Krämer N & Petschelt A (1998) Bond strengths of luting composites to IPS Empress after silica coating *Journal of Dental Research* **77** Abstract of Papers p 678 Abstract 369.

Bronnimann R, Fritzsche T & Schärer P (1991) Porcelain repairs. A review of the literature and practical tips for porcelain repairs on VMK (metal-ceramic) reconstructions (Abstract in English) *Schweizerische Monatsschrift für Zahnmedizin* **101(6)** 762-773.

Chen J-H, Matsumura H & Atsuta M (1997) Effect of different etching periods on the bond strength of a composite resin to a machinable porcelain *Journal of Dentistry* **26(1)** 53- 58.

Chung KH & Hwang YC (1997) Bonding strengths of porcelain repair systems with various surface treatments *Journal of Prosthetic Dentistry* **78(3)** 267-274.

Della Bona A & van Noort R (1995) Shear vs Tensile bond strength of resin composite bonded to ceramic *Journal of Dental Research* **74(9)** 1591-1596.

Hummel SK, Pace LL & Marker VA (1994) A comparison of two silicoating techniques *Journal of Prosthodontics* **3(2)** 108-113.

Ishijima T, Caputo AA & Mito R (1992) Adhesion of resin to casting alloys *Journal of Prosthetic Dentistry* **67(4)** 445-449.

Kelly JR, Nishimura I & Campbell SD (1996) Ceramics in dentistry: historical roots and current perspectives *Journal of Prosthetic Dentistry* **75(1)** 18-32.

Kupiec KA, Wuertz KM, Barkmeier WW & Wilwerding TM (1996) Evaluation of porcelain surface treatments and agents for composite-to-porcelain repair *Journal of Prosthetic Dentistry* **76(2)** 119-124.

Matsumura H & Atsuta M (1996) Repair of an eight-unit fixed partial denture with a resin-bonded overcasting: a clinical report *Journal of Prosthetic Dentistry* **75(6)** 594-596.

Phoenix RD & Shen C (1995) Characterization of treated porcelain surfaces via dynamic contact angle analysis *International Journal of Prosthodontics* **8(2)** 187-194.

Rada RE (1991) Intraoral repair of metal ceramic restorations *Journal of Prosthetic Dentistry* **65(3)** 348-350.

Schneider W, Powers JM & Pierpont HP (1992) Bond strength of composites to etched and silica-coated porcelain fusing alloys *Dental Materials* **8** 211-215.

van der Vyver PJ, Marais JT & de Wet FA (1996) Repair of Cerec porcelain with seven different systems *Journal of the Dental Association of South Africa* **51** 525-520.

Wolf DM, Powers JM & O'Keefe KL (1992) Bond strength of composite to porcelain treated with new porcelain repair agents *Dental Materials* **8** 158-161.

Zhukovsky L, Godder B, Settembrini L & Scherer W (1996) Repairing porcelain restorations intraorally: techniques and materials *Compendium of Continuing Education in Dentistry* **17(1)** 18-30.

©Operative Dentistry, 2000, **25,** 216-222

Factors Affecting Shear Bond Strength of Composite Resin to Fluorosed Human Enamel

N Ateyah • E Akpata

Clinical Relevance

Age affects shear bond strength of composite resin to fluorosed teeth; when fluorosis is severe, bond failure is most likely to be cohesive in enamel.

SUMMARY

The aim of this work was to determine the effects of age, severity of fluorosis, and etching time on the shear bond strength of direct composite resin to human enamel. A total of 117 teeth, freshly extracted from patients in areas of Saudi Arabia endemic for dental fluorosis, were classified according to age (<40 years and 40+ years) and severity of fluorosis, using the Thylstrup and Fejerskov index, TFI: TFI = 0, TFI = 1-3, and TFI = 4-6. Cylindrical composite resin specimens 5 mm in diameter and 3 mm high were bonded to the flattened midlabial enamel surfaces etched for 60 or 120 seconds and shear bond strength measured, using the Instron Universal Testing Machine at a crosshead speed of 0.5 mm/ minute.

Shear bond strength of the resin varied between 11.2±3.6 and 21.6±4.1 MPa. Three-way analysis of variance and Sheffé's multiple range test showed that the severity of fluorosis had no statistically significant effect on shear bond strength (p >0.05). However, the bond was significantly stronger in teeth from patients <40 years old than from those 40+ years old. Furthermore, at age <40 years, shear bond strength was significantly higher in teeth etched for 120 seconds than those etched for 60 seconds (p<0.05), but this was not the case in teeth from the older patients. In teeth with TFI = 1-3, the mode of bond failure was predominantly mixed, but at TFI = 4-6, the bond failure was mostly cohesive in enamel at all ages and etching times. It is, therefore, concluded that both age and etching time affect the shear bond strength of composite resin to fluorosed human enamel.

Department of Restorative Dental Sciences, King Saud University College of Dentistry, P.O. Box 60169, Riyadh, 11545, Saudi Arabia

Nasreen Ateyah

ES Akpata, professor

INTRODUCTION

Enamel fluorosis is characterized by surface hypermineralization and porosity of the subsurface layer (Thylstrup & Fejerskov, 1978). The hypermineralized surface enamel is difficult to acid etch (Opinya & Pameijer, 1986), while the porous subsurface layer may attract extrinsic stains posteruptively, resulting in tooth discoloration (Fejerskov, Manji & Baelum, 1990). In severe cases of dental fluorosis, the subsurface porosity is so extensive that occlusal trauma may give rise to the detachment and pitting of surface enamel (Thylsrup & Fejerskov, 1978). The discolored and pitted enamel may be esthetically objectionable, sometimes resulting in psychological ill health.

Dental fluorosis is endemic in several parts of the world (Dean, Arnold & Evolve, 1942; Leverett, 1986; Warnakulasuriya & others, 1992; Akpata, Fakiha & Khan, 1997), and the use of fluoride in preventive dentistry over the past few decades has resulted in increased prevalence of the condition in many countries (Leverett, 1986). One of the treatment modalities for moderately or severely fluorosed teeth is the provision of the composite resin veneer (Akpata, 1997). Bonding of the veneer involves etching the acid-resistant fluorosed enamel, and this may necessitate prolonging the etching time.

It was hypothesized that the bond strength of composite resin to fluorosed enamel would be influenced by the severity of fluorosis, etching time, and age of the patient. The purpose of this study, therefore, was to investigate the effects of age, severity of fluorosis, and etching time on the shear bond strength of composite resin to human enamel.

METHODS AND MATERIALS

Diagnosis of Dental Fluorosis

The diagnosis of fluorosis was practiced on 46 clinical slides of fluorosed teeth classified according to the modified Thylstrup and Fejerskov index, TFI (Fejerskov, Manji & Baelum, 1988) by one of the investigators (NA). The diagnosis was repeated after two weeks; the test of intraexaminer reproducibility gave a Cohen's kappa statistic (Cohen, 1960) of 0.974. In addition, both investigators carried out the diagnoses independently, and a test of interexaminer reproducibility resulted in a Cohen's kappa statistic (Cohen, 1960) of 0.972.

Collection and Grouping of Experimental Teeth

A total of 117 fluorosed and nonfluorosed anterior and posterior teeth (12 incisors, 69 premolars, and 36 molars) were used in this study. These were obtained from Hail and Al-Ras, two areas of Saudi Arabia endemic for dental fluorosis. The teeth had no obvious damage as a result of extraction, caries, or abrasion. The extracted teeth were cleaned with an ultrasonic scaler, polished with a brush and nonfluoride flour of pumice, and then stored in distilled water containing 0.2% thymol as a disinfectant, in a refrigerator. The teeth were kept in two separate jars according to the age of the patient from whom they were extracted: < 40 years and 40+ years of age.

Prior to use, the experimental teeth in each age group were classified according to the severity of fluorosis, using the TFI: TFI = 0, TFI = 1-3, and TFI = 4-6. The distribution of teeth in each group is shown in Figure 1.

Preparation of Enamel Surface

To standardize the enamel reduction, depth orientation grooves 0.5 mm deep were prepared on the midlabial surfaces of each of the experimental teeth. The teeth were then mounted in autopolymerizing acrylic resin in standardized PVC molds 20 mm high, with an internal diameter of 26 mm. The grooved midlabial surfaces of the experimental teeth were then flattened with 240- and 600-grit silicon carbide paper.

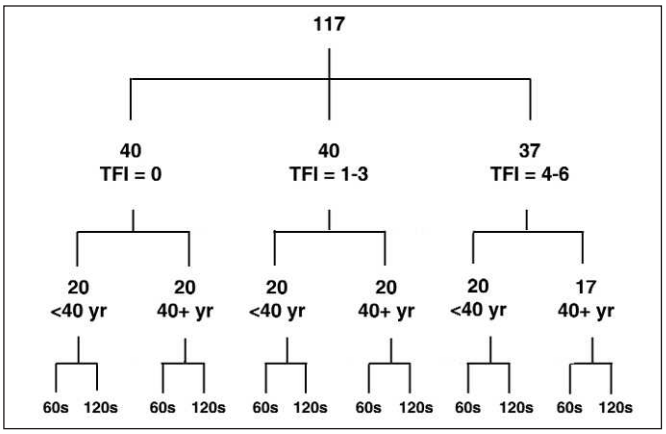


Figure 1. *Grouping of teeth.*

The flattened midlabial surface of each tooth specimen was polished with a rubber cup and nonfluoride flour of pumice for 20 seconds, washed with distilled water for 20 seconds, and dried with an air syringe for an additional 20 seconds. Air from the syringe was filtered with a single filter (AF100; Yoshida Works, Osaka, Japan).

Etching

Coltène Etchant Gel (Brilliant Esthetic System, Batch No EJ435; Coltène/Whaledent Inc, Mahwah, NJ 07430) containing 35% phosphoric acid was applied with a brush to cover the flattened enamel surface of each specimen. Half of the specimens in each group were etched with the phosphoric acid for 60 seconds and the other half for 120 seconds. However, in the group of teeth with TFI = 4-6 from patients aged 40+ years, nine teeth were etched for 60 seconds and the remaining eight teeth for 120 seconds (Figure 1). The etched surfaces were then washed with a gentle stream of distilled water for 20 seconds and dried with filtered air from the syringe for 20 seconds.

Application of Bonding Resin and Composite Resin

Coltène Margin Bond (Brilliant Esthetic System, Batch No EJ435), an enamel bonding agent, was applied with a brush to the etched enamel surface of each specimen. The bonding agent was lightly air sprayed to produce a thin film and cured for 20 seconds with a light-curing unit (Coltolux 4; Coltène/ Whaledent). The power output of the curing unit was checked periodically to ensure that it was not less than 300 mW/cm^2 (Barghi, Berry & Hatton, 1994).

Coltène Brilliant Enamel hybrid composite resin, shade A2 (Brilliant Esthetic System, Batch No RJ435) was applied to the etched midlabial surface of each specimen in a cylindrical split Teflon mold 3 mm high, with an internal diameter of 5 mm. The mold was seated firmly at the center of the flattened tooth surface. After curing the composite resin for 60 seconds, the mold was removed. The specimen was then stored at 37°C in distilled water for 24 hours before shear bond testing.

Shear Bond Strength Measurement

Bond strength between the composite resin and enamel was measured in shear mode with an Instron Universal Testing Machine (Model 1197; Instron Corp, Buckinghamshire, England). The specimen was mounted in a fixture and a knife-edge loading device applied to the composite resin specimen as close to its junction with enamel as possible at a crosshead speed of 0.5 mm per minute. Fracture loads were recorded in newtons on a strip chart. The recorded loads were converted to MPa by dividing the loads by the cross-sectional area of the composite resin cylinder.

Mode of Bond Failure

The mode of failure of the bond between the composite resin and enamel was determined using the light microscope at X10 magnification and classified into adhesive, cohesive, and mixed types of failure (Nakajima & others, 1995). Adhesive failure was recorded when there were no signs of enamel fracture or remnants of resin on the tooth, cohesive fracture when there was complete fracture of enamel or resin, and mixed failure when the samples showed both adhesive and cohesive failures. In addition, 40 representative specimens from the 12 sample subgroups that included those with adhesive, cohesive, and mixed modes of failure were selected for scanning electron microscopic study. The specimens were thoroughly washed with distilled water, dried, and secured onto metal stubs with colloidal carbon adhesive. They were then sputter coated with gold and examined at x1000-2000 magnification under the scanning electron microscope (JEOL SMT 330; JEOL Ltd, Tokyo, Japan) at 15 kv. Photomicrographs were taken using Kodak Verichrome Pan 120mm black-and-white negative films, VP 120.

Statistical Analysis

Statistical significance among shear bond strength, TFI, age, and etching time was determined at a 5% probability level using the three-way analysis of variance (ANOVA) with interaction, as well as Scheffé's multiple range test.

Table 1: *Mean shear bond strength in MPa (± standard deviation) of composite resin to enamel with varying severity of fluorosis after etching for 60 or 120 seconds (shear bond strength values with different letters in superscript indicate statistically significant differences).*

	TFI	Etching 60 s.	Time 120 s.
Age <40 yr.	0	18.2 (7.1)^{a}	20.8 (2.9)^{c}
	1-3	19.3 (5.3)^{a}	21.6 (3.0)^{c}
	4-6	18.8 (5.2)^{a}	21.6 (4.1)^{c}
Age 40+ yr.	0	10.7 (2.1)^{b}	12.4 (3.0)^{b}
	1-3	19.0 (6.1)^{d}	12.9 (6.1)^{b}
	4-6	14.2 (7.3)^{bd}	11.2 (3.6)^{b}

RESULTS

Teeth

Each sample subgroup of 10 teeth (Figure 1) comprised one incisor, six premolars, and three molars, except the two groups with TFI = 4-6 from patients aged 40+ years that consisted of five and four premolars, respectively.

Shear Bond Strength

The three-way ANOVA and Sheffé's multiple range test showed that the severity of fluorosis had no significant effect on shear bond strength of composite resin to enamel ($p>0.05$). The only exception were the teeth with TFI = 1-3, which had significantly stronger bond than those with TFI = 4-6 at age 40+ years ($p<0.05$). However, the bond strength was significantly higher in teeth extracted from patients aged <40 years than in those aged 40+ years ($p<0.05$) (Table 1).

At age <40 years, shear bond strength was significantly higher in teeth etched for 120 seconds than in those etched for 60 seconds ($p<0.05$), but this was not the case in teeth extracted from patients aged 40+ years ($p>0.05$) (Table 1).

The coefficient of variation of the shear bond strength measurement ranged from 14-51%, and the power of the test was approximately 70%.

Mode of Bond Failure

In nonfluorosed teeth etched for 60 seconds, adhesive mode of failure was most predominant in teeth from patients aged <40 years. However, when etching time increased from 60 to 120 seconds, more cases of mixed failure became evident (Tables 2-3). In teeth with TFI = 1-3, the mode of failure was predominantly mixed at all ages and etching times. At TFI = 4-6, the mode of failure was mostly cohesive in enamel when etched for 60 or 120 seconds at all ages. There were no cases of cohesive failure in composite resin (Tables 2-3).

Table 2: *Frequency of teeth with varying severity of fluorosis at age >40 years according to the mode of failure of bonded composite resin.*

Mode of Failure	TF=0		TFI=1-3		TFI=4-6		Total
	60 sec.	120 sec.	60 sec.	120 sec.	60 sec.	120 sec.	
Cohesive in enamel	1	1	4	2	6	4	18
Cohesive in composite	0	0	0	0	0	0	0
Adhesive	5	2	2	0	4	0	13
Mixed	4	7	4	8	0	6	29
Total	10	10	10	10	10	10	60

Table 3: *Frequency of teeth with varying severity of fluorosis at age 40+ years according to the mode of failure of bonded composite resin.*

Mode of Failure	TF=0		TFI=1-3		TFI=4-6		Total
	60 sec.	120 sec.	60 sec.	120 sec.	60 sec.	120 sec.	
Cohesive in enamel	3	0	0	0	5	6	14
Cohesive in composite	0	0	0	0	0	0	0
Adhesive	3	2	2	2	0	0	9
Mixed	4	8	8	8	4	2	34
Total	10	10	10	10	9	8	57

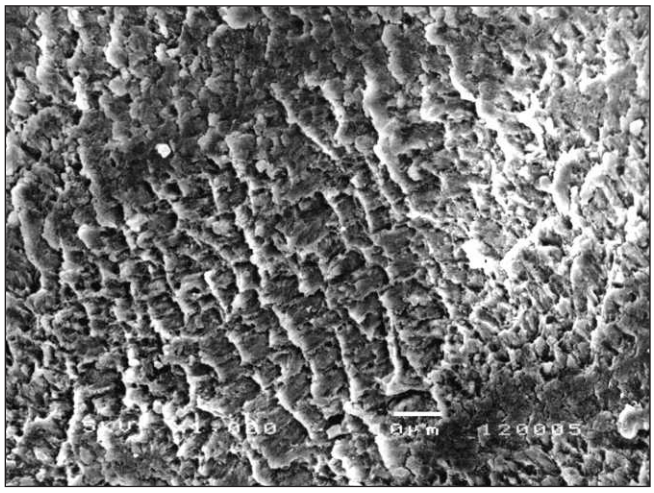


Figure 2. *Scanning electron micrograph showing enamel surface after adhesive failure on a tooth with TFI=0 at age <40 years and etching time of 60 seconds (original magnification x1000).*

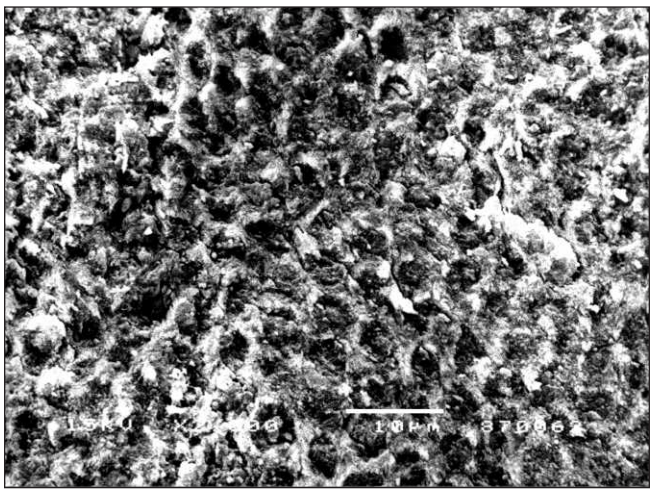


Figure 3. *Scanning electron micrograph showing enamel surface after adhesive failure on a tooth with TFI=1-3 at age 40+ years and etching time of 120 seconds (original magnification x2000).*

In cases with adhesive mode of failure, SEM examination revealed typical etching patterns (Figures 2 and 3), especially in the nonfluorosed teeth. In teeth with TFI = 4-6, exposed dentinal tubules could be seen in some cases with cohesive or mixed failure (Figure 4), suggesting detachment of enamel at the dentinoenamel junction. The small sample size did not permit statistical analysis for the significance of the differences between the different modes of failure.

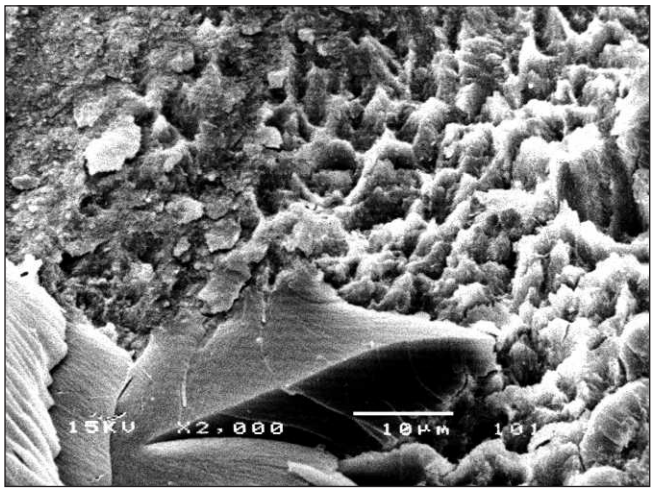


Figure 4. *Scanning electron micrograph showing enamel surface after mixed failure on a tooth with TFI=4-6 at age 40+ years and etching time of 120 seconds. Some open ends of dentinal tubules are visible on the top left corner (original magnification x2000).*

DISCUSSION

The classification of fluorosis in this study was according to the modified Thylstrup and Fejerskov index (Fejerskov & others, 1988), which is based on the clinical changes in fluorosed teeth. The main advantage of this classification is that it is consistent with the histopathological changes in fluorosed enamel. Moreover, the index is highly reproducible, as evidenced by the high kappa stastistics (Cohen, 1960) of 0.972-0.974 obtained from inter- and intraexaminer reproducibility tests in the present study.

Teeth with TF I = 1-3 were grouped together because Al-Sugair and Akpata (1999) have shown that the pattern and depth of etch were similar within this group. Furthermore, Opinya and Pameijer (1986) reported that tensile bond strength of resin composite to teeth with TFI = 1-3 was similar.

As the number of teeth available for the study was limited, we decided to divide the samples into only two age groups: <40 years and 40+ years. This decision was based on the fact that most teeth tend to be lost because of periodontal disease after age 40 (Reich & Hiller, 1993; Murray, Locker & Kay, 1996). Thus the chances of finding caries-free teeth for the study was increased. The effect that different age groups might have on the results may be elucidated by future research.

Knoll, Gwinnett, and Wolff (1986) reported statistically significant differences in the shear strength of orthodontic brackets bonded to anterior and posterior teeth. They attributed the lower values in posterior teeth to adaptation and nonuniform resin thickness, rather than differences in etching pattern. To minimize the effect of tooth types in the present study, each sample subgroup comprised approximately equal proportions of incisors, premolars, and molars. Furthermore, bonding was to the flattened midlabial surfaces of the teeth.

As far as we know, there are no reports of previous studies on the shear bond strength of composite resin to fluorosed enamel. Consequently, the predetermination of the sample size was not easy. Each of our sample subgroups comprised approximately 10 teeth, as in several previous studies utilizing nonfluorosed teeth (Gilpatrick, Ross & Simonsen, 1991; Hadavi & others, 1993), and each of the fluorosis sample groups consisted of approximately 40 teeth (Figure 1). Moreover, the coefficient of variation obtained (14-51%) in the present study is within the range reported in the literature (Barkmeier, Shaffer & Gwinnett, 1986; Opinya & Pameijer, 1986; Guba, Cochran & Swartz, 1993; Holtan & others, 1995). As the power of the test was approximately 70%, the sample size, in an investigation of this nature, may be regarded as reasonably adequate. The use of the same tooth types in future studies may reduce the variability in the shear bond strength and also increase the power of the test.

Opinya and Pameijer (1986), in their study of tensile bond strength of composite resin to fluorosed teeth, etched enamel for 120 seconds. However, Al-Sugair and Akpata (1999), in their investigation of the etching pattern of fluorosed teeth, recommended an etching time of at least 30 seconds for fluorosed teeth with TFI = 4 and at least 90 seconds for more severely fluorosed teeth with pitting (TFI = 5+). Therefore in the present study, etching times of 60 (mean of 30 and 90) and 120 seconds were used.

Richards, Fejerskov, and Baelum (1989) showed that the highest concentration of fluoride is in the outer 200 µm of fluorosed enamel. This outer hypermineralized layer is highly resistant to acid etching (Al-Sugair & Akpata, 1999). Opinya and Pameijer (1986) observed that grinding away this outer hypermineralized layer before etching resulted in higher tensile bond strength. In the present study, the outer 0.5 mm of enamel was ground away to flatten the enamel surface for shear bond strength measurements. This is consistent with clinical practice when the outer 0.5 mm of labial enamel is removed during tooth preparation for composite resin or porcelain laminate veneer. On the other hand, bond strength values might have been lower if the composite resin were bonded to the hypermineralized enamel surfaces of the fluorosed teeth, without grinding. There is a need to develop a technique for measuring shear bond strength of composite resin to fluorosed enamel without grinding to flatten the hypermineralized surface layer. The result from such an investigation would be useful to clinicians who place laminate veneers without tooth preparation.

The shear bond strength obtained in this study would be expected to be similar to initial bond of direct composite resin veneer. This is because bond strength (in MPa) is independent of the thickness of the composite resin used for bond strength testing, provided the loading plane is as near the interface as possible. Besides, there appears to be no method available for the direct measurement of purely shear bond strength of composite resin veneer to enamel. As the restoration ages, however, the bond may deteriorate. The procedures most often used to simulate aging are long-term water storage at a constant temperature or thermocycling. The validity of thermocycling has been questioned by many investigators (Crim & Mattingly, 1981; Brännström, 1984; Eackle, 1986), who were of the opinion that the experimental procedure may change the properties of the restorative material and hardly simulates clinical situations. In the present study, prior to bond testing, we stored the specimens in water at 37°C for 24 hours.

The studies on tensile bond strength of composite resin to fluorosed teeth reported by Opinya and Pameijer (1986) as well as Ng'ang'a and others (1992) were aimed at determining dislodging forces on orthodontic brackets, and these are predominantly tensile. In the present study, we measured shear bond strength because it more closely approximates to the dislodging forces acting on composite resin veneers.

The shear bond strength of 18±7.1 MPa obtained for teeth with TFI = 0 (ie, nonfluorosed teeth) etched for 60 seconds in this study is rather similar to about 20 MPa reported for nonfluorosed teeth by other investigators (Barkmeier & others, 1986; Gilpatrick & others, 1991; Gwinnett, 1992). In fact, for teeth with TFI = 4-6 in the present study, shear bond strength was as high as 21.6±4.1 MPa at age <40 years (Table 1). In general, however, severity of fluorosis per se did not have a statistically significant effect on shear bond strength (Table 1). This is in agreement with the report by Ng'ang'a and others (1992), who observed no significant difference between the mean tensile bond strengths in teeth with TFI = 0, 3, or 4.

Posteruptive trauma weakens the surface enamel in severely fluorosed teeth (TFI = 5+), resulting in its detachment and, therefore, surface pitting (Thylstrup & Fejerskov, 1978). Although there is no pitting at TFI = 4, subsurface porosity is quite extensive, and the surface enamel could still be weakened with advancing age. This weakening of surface enamel may explain the higher prevalence of cohesive mode failures in enamel in teeth with TFI = 4-6. It may also explain why increasing etching time from 60 to 120 seconds did not result in a significant increase in the shear bond of the composite resin to severely fluorosed teeth (TFI = 4-6) of patients aged 40+ years (Table 1). However, the effect of age on the bond to enamel of nonfluorosed teeth needs to be investigated by future research. The exposure of dentinal tubules in some cases with mixed mode of failure or cohesive failure in enamel is consistent with clinical experience: enamel tends to shell off in some severely fluorosed teeth. Further research is needed to elucidate the effect of fluorosis on the dentinoenamel junction.

In clinical practice, therefore, a resin composite veneer is likely to be more retentive when etching time is increased from 60 to 120 seconds in patients aged below 40 years, but not in older patients. Furthermore, composite resin veneers are more likely to be debonded from severely fluorosed teeth (TFI = 4+) due to cohesive bond failure in enamel, especially in those patients aged 40+ years.

CONCLUSIONS

The severity of fluorosis has no significant effect on shear bond strength of composite resin to enamel. However, increasing etching time from 60 to 120 seconds resulted in a significant increase in shear bond strength for teeth of patients aged <40 years. Bond failure in severely fluorosed teeth was most likely to be cohesive in enamel.

Acknowledgment

This work was done in partial fulfilment of the requirements for the Master's degree of the King Saud University College of Dentistry, Riyadh, Saudi Arabia. We are grateful to the College of Dentistry Research Center for providing the facilities for this work.

(Received 29 December 1998)

References

Akpata E S (1997) *A Textbook of Operative Dentistry* London: Class Publishing p 97.

Akpata ES, Fakiha Z & Khan N (1997) Dental fluorosis in 12-15-year-old rural children exposed to fluorides from well drinking water in the Hail region of Saudi Arabia *Community Dentistry and Oral Epidemiology* **25(4)** 324-327.

Al-Sugair MH & Akpata ES (1999) Effect of fluorosison on etching of human enamel *Journal of Oral Rehabilitation* **26(6)** 521-528.

Barghi N, Berry T & Hatton C (1994) Evaluating intensity output of curing lights in private dental offices *Journal of the American Dental Association* **125(7)** 992-996.

Barkmeier WW, Shaffer SE & Gwinnett AJ (1986) Effects of 15 vs 60 second enamel acid conditioning on adhesion and morphology *Operative Dentistry* **11(3)** 111-116.

Brännström M (1984) Communication between the oral cavity and the dental pulp associated with restorative treatment *Operative Dentistry* **9(2)** 57-68.

Cohen J (1960) A coefficient of agreement for nominal scales *Educational and Psychological Measurement* **20** 37-46.

Crim GA & Mattingly SL (1981) Evaluation of two methods of assessing marginal leakage *Journal of Prosthetic Dentistry* **45(2)** 160-163.

Dean HT, Arnold FA & Evolve E (1942) Domestic water and dental caries. V. Additional studies of the relation of fluoride in domestic waters to dental caries in 4,425 white children, aged 12-14 years of 13 cities in 4 states *Public Health Report* **57** 1155-1179.

Eakle WS (1986) Effect of thermal cycling on fracture strength and microleakage in teeth restored with a bonded composite resin *Dental Materials* **2(3)** 114-117.

Fejerskov O, Manji F & Baelum V (1988) *Dental Fluorosis–a Handbook for Health Workers* Copenhagen: Munksgaard p 45.

Fejerskov O, Manji F & Baelum V (1990) The nature and mechanism of dental fluorosis in man *Journal of Dental Research* **69** (Special Issue) 692-700.

Gilpatrick RO, Ross JA & Simonsen RJ (1991) Resin-to-enamel bond strengths with various etching times *Quintessence International* **22(1)** 47-49.

Guba CJ, Cochran MA & Swartz ML (1994) The effect of varied etching time and etching solution viscosity on bond strength and enamel morphology *Operative Dentistry* **19(4)** 146-153.

Gwinnett AJ (1992) Structure and composition of enamel *Operative Dentistry* **Supplement 5** 10-17.

Hadavi F, Hey JH, Ambrose ER, Louie PW & Shinkewski DJ (1993) The effect of dentin primer on the shear bond strength between composite resin and enamel *Operative Dentistry* **18(2)** 61-65.

Holtan JR, Nystrom GP, Phelps RA, Anderson TB & Becker WS (1995) Influence of different etchants and etching times on shear bond strength *Operative Dentistry* **20(3)** 94-99.

Knoll M, Gwinnett AJ, Wolff MS (1986) Shear strengths of brackets bonded to anterior and posterior teeth *American Journal of Orthodontics* **89(6)** 476-479.

Leverett D (1986) Prevalence of dental fluorosis in fluoridated and nonfluoridated communities—a preliminary investigation *Journal of Public Health Dentistry* **46(4)** 184-187.

Murray H, Locker D & Kay EJ (1996) Patterns and reasons for tooth extractions in general dental practice in Ontario, Canada *Community Dentistry and Oral Epidemiology* **24(3)** 196-200.

Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM & Pashley DH (1995) Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesives *Journal of Dental Research* **74(10)** 1679-1688.

Ng'ang'a PM, Øgaard B, Cruz R, Chindia ML & Aastrum M E (1992) Tensile strength of orthodontic brackets bonded directly to fluorotic and nonfluorotic teeth: an *in vitro* comparative study *American Journal of Orthodontics and Dentofacial Orthopedics* **102(3)** 244-250.

Opinya GN & Pameijer CH (1986) Tensile bond strength of fluorosed Kenyan teeth using the acid etch technique *International Dental Journal* **36(4)** 225-229.

Reich E & Hiller KA (1993) Reasons for tooth extraction in the western states of Germany *Community Dentistry and Oral Epidemiology* **21(6)** 379-383.

Richards A, Fejerskov O & Baelum V (1989) Enamel fluoride in relation to severity of human dental fluorosis *Advances in Dental Research* **3(2)** 147-153.

Thylstrup A & Fejerskov O (1978) Clinical appearance of dental fluorosis in permanent teeth in relation to histological changes *Community Dentistry and Oral Epidemiology* **6(6)** 315-328.

Warnakulasuriya KA, Balasuriya S, Perera PA & Peiris LC (1992) Determining optimal levels of fluoride in drinking water for hot, dry climates—a case study in Sri Lanka *Community Dentistry and Oral Epidemiology* **20(6)** 364-367.

©Operative Dentistry, 2000, **25,** 223-227

Rigidity and Retention of Ceramic Root Canal Posts

DG Purton • RM Love • NP Chandler

Clinical Relevance

Tapered, smooth ceramic root canal posts were more rigid but less retentive in roots than similar-sized parallel-sided, serrated stainless steel posts.

SUMMARY

Ceramic root-canal posts offer potential advantages over other types with respect to aesthetics and biocompatibility. Any post must be sufficiently rigid and retentive to withstand functional forces. Ceraposts (1.2 mm coronal diameter, ceramic, tapering, smooth posts) and Paraposts (1.25 mm, stainless-steel, parallel, serrated posts) were tested for rigidity by means of a three-point bending test. To test retention in roots, ceramic posts were cemented using one of three protocols: (1) glass-ionomer cement, (2) silane coupling agent and resin cement, or (3) sandblasted post surface, silane coupling agent, and resin cement. Stainless-steel posts were cemented with resin. The tensile force required to dislodge the posts, following four weeks of storage in water, was recorded. Data were compared using Student's t-test and Mann-Whitney U analysis. Ceraposts were significantly more rigid than Paraposts ($p<0.001$). Paraposts cemented with resin were significantly more strongly retained than Ceraposts following any cementation protocol ($p<0.001$). Retention of the ceramic posts was significantly greater with a silane coupling agent and resin cement than with glass-ionomer cement ($p<0.001$). Sandblasting the ceramic posts produced variable results and needs further investigation before it could be recommended.

University of Otago, Department of Oral Rehabilitation, School of Dentistry, 280 Great King Street, PO Box 647, Dunedin, New Zealand

David G Purton, senior lecturer

Robert M Love, senior lecturer

NP Chandler, senior lecturer

INTRODUCTION

Parallel-sided, serrated, stainless-steel posts, typified by the Parapost, have a long record of clinical success, and in many countries represent the industry standard for prefabricated posts (Schwartz, Summitt & Robbins, 1996; Torbjörner, Karlsson & Ödman, 1995). Issues of aesthetics and biocompatibility have led dentists and patients to increasingly demand metal-free restorations. Consequently, there has been progress in the search for durable, aesthetic, biocompatible ceramic and resin restorations.

The excellent biocompatibility of ceramics has been established for some time. In addition, many ceramic restorative materials have a high degree of translucency (Anusavice, 1996). This can allow light transmission into the underlying tooth tissue, thereby avoiding the root darkening that commonly occurs in teeth restored with opaque materials.

Historically, the disadvantage of ceramic materials has been their low flexural strength compared with metals.

In function, ceramic restorations have a record of frequent failure in high-stress situations (Anusavice, 1996). Recently developed ceramic materials consisting of zirconium dioxide stabilized with small amounts of yttrium oxide offer high flexural strength and toughness (Christel & others, 1989; Shimizu & others, 1993). Root-canal posts made of these materials are commercially available and offer the potential for post and core restorations with adequate strength for normal function and the advantage of superior aesthetics to current alternative systems. A clinical trial has been reported in which 80 ceramic posts with composite resin or ceramic cores were observed over 16.6 ± 9.1 months. No posts were fractured and none were lost to debonding (Kern, Simon & Strub, 1998).

Although the ceramic and stainless steel posts are quite different products, either post could be chosen in a given clinical situation, making comparisons between the two clinically relevant. Therefore, the purposes of this study were (1) to compare the rigidity of ceramic root-canal posts with stainless-steel posts of similar size, and (2) to compare the retention in roots of ceramic posts using three different cementation regimens with the retention of stainless-steel posts.

METHODS AND MATERIALS

Rigidity Test

The ceramic posts tested (Cerapost; Gebr Brasseler, Lemgo, Germany) are made of 94.9% zirconium dioxide (ZrO_2), stabilized with 5.1% yttrium oxide (Y_2O_3). Ten size 50 ceramic posts, which measure 0.5 mm in diameter at the apical tip and 1.2 mm in diameter in the coronal portion, were tested for rigidity using a three-point bending test in a Universal Testing Machine (Instron, High Wycombe, UK). The test is a variation of the ASTM standard method, designation E 855-84. The posts were supported as shown in Figure 1 with the parallel-sided coronal portion spanning the test jig, and the load applied at the mid-point. The crosshead speed of the testing machine was 5 mm per minute, and the load was applied until the posts fractured. Ten 1.25 mm stainless-steel, serrated posts (Parapost; Coltène/Whaledent Inc, Mahwah, NJ 07430) were tested in a similar manner. Loading of these posts continued until they reached their yield point, as indicated by the force/deflection curve becoming nonlinear.

All of the data were recorded in an Apple Macintosh computer using MacLab Chart software. A plot of force (N) versus deflection (μm) was made for each post. The gradients of the plots for the two groups of posts were compared using Student's *t*-tests. The mean gradient for each post type was calculated and used for graphic depiction of the comparison.

Retention Test

Forty single-rooted human teeth, free of defects or restorations in the roots and with narrow, unfilled root canals, were selected for use in the study. The teeth were sectioned horizontally 1 mm above the labial cemento-enamel junction. The roots were grooved on their outside surfaces using a diamond bur in a high-speed handpiece, embedded in individual acrylic blocks with the root face exposed, and stored in sterile, deionized water at room temperature until tested.

The root canals were prepared using the step-down method (Goerig, Michelich & Schultz, 1982) with 2.5% sodium hypochlorite irrigation. This is a modification of the step-back technique, resulting in a flared preparation with minimal apical enlargement. The canals were filled with laterally-condensed gutta-percha and AH26 sealer (Dentsply DeTrey Division, Dentsply Ltd, Weybridge, Surrey, UK). The roots were randomly assigned to four groups of 10. For all groups post-canal preparations were made to a depth of 10 mm by removing gutta-percha with Gates-Glidden drills (Dentsply Maillefer, Ballaigues, Switzerland) and preparing the canals with the post-hole drills supplied by the manufacturer of the posts to be cemented.

In Group 1 the dentin of the post canals was etched for 15 seconds with 37% phosphoric acid gel, rinsed with water for 30 seconds, and dried with paper points. The stainless-steel posts were cleaned with isopropyl alcohol and air dried. Each post was cemented to full depth with freshly mixed resin cement (Flexi-Flow Natural; Essential Dental Systems, Hackensack, NJ 07606) introduced into the canal by a Pastinject spiral filler (Micro Mega, Besancon, France), and held in position for 15 minutes.

Groups of ceramic posts were cemented using one of the three alternative cementing regimens suggested by the manufacturer.

In Group 2 the root portion of the ceramic posts was sandblasted by rotating the post for 10 seconds in a stream of 50 μm aluminium oxide. They were cleaned with isopropyl alcohol, air dried, and coated with silane primer (3M Scotchbond Ceramic Primer; 3M Dental Products, St Paul, MN 55144) used according to the manufacturer's instructions. The dentin of the post canal was instrumented with the diamond roughening instrument supplied by the manufacturer. Dentin etching, rinsing, drying, and cementation were the same as for Group 1.

In Group 3 the ceramic posts were cleaned with isopropyl alcohol, air dried, and silane coated. Dentin roughening was the same as for Group 2. Etching, rinsing, drying, and cementation were the same as for Groups 1 and 2.

In Group 4 the dentin of the post canals was rinsed with water and dried with paper points. The ceramic posts were cleaned with isopropyl alcohol, air dried, and cemented to full depth with freshly mixed capsulated glass-ionomer cement (Fuji 1; GC Corp, Tokyo, Japan) and held in place for 15 minutes.

Following storage of all specimens in sterile, deionized water at room temperature for four weeks, the portions of the ceramic posts extending from the roots had pieces

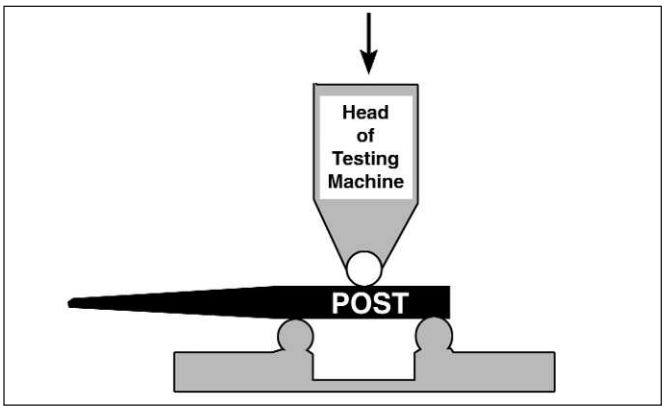


Figure 1. *Diagram of a ceramic post undergoing the three-point bending test.*

of close fitting, stainless-steel tube cemented to them to provide protection from crushing in the vice of the testing machine. Each specimen was then placed into a retention device and mounted in the testing machine. Figure 2 shows the arrangement of post, root, acrylic block, and retention testing device. A universal coupling in the testing machine ensured that the load was directed axially along the post. The crosshead speed of the testing machine operated at 5 mm per minute, and the tensile force (N) required to remove the post from the root was recorded using the computer software as described above. The data for each group were statistically compared with the other groups using Student's *t*-tests and Mann-Whitney U tests.

The ceramic posts were inspected visually after testing to determine the mode of failure, by looking for traces of cement on their surfaces.

Electron Microscopy

Scanning electron microscopy (SEM) was used to examine the surface topography of the posts as supplied by the manufacturer, following sandblasting, and the fractured surfaces of five posts chosen at random following the three-point bending tests. Specimens were mounted onto SEM stubs with silver paste and prepared for examination by gold coating in a Poloron E5100 coating unit (Poloron Equipment Ltd, Watford, UK). Representative black-and-white photographs were taken at various magnifications when viewed with a Cambridge stereoscopic 360SEM (Cambridge Instruments Co, Cambridge, UK).

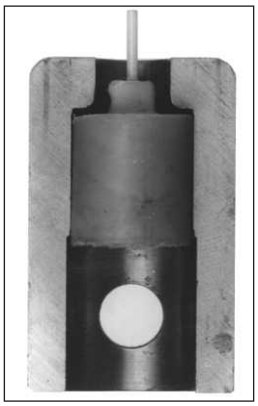


Figure 2. *A ceramic post, root, and acrylic block shown in one-half of the retention device.*

RESULTS

Rigidity Test

The force versus deflection gradients for the 10 ceramic posts and the 10 stainless-steel posts (Table 1) were compared using Student's *t*-test. This revealed that the ceramic posts were significantly more rigid than the stainless-steel posts ($p<0.001$). Figure 3 shows the mean gradient for the ceramic posts (1.96 ± 0.2) and for the stainless-steel posts (0.18 ± 0.03). The highest points on the two plots represent the force and deflection values at the yield points of the posts. At these points the ceramic posts fractured and the stainless-steel posts became permanently deformed.

Retention Test

The mean retention values for the four groups of posts are shown in Table 2. Stainless-steel posts cemented with resin cement were more strongly retained than ceramic posts following any cementation protocol ($p<0.001$). The only other significant result was that the ceramic posts cemented with glass-ionomer cement were less strongly retained than when cemented with resin ($p<0.001$). Some of the sandblasted posts fractured during the retention tests.

Table1: *Rigidity of Posts Determined by Force/Deflection (Gradient)*

Ceramic Posts			**Stainless Steel Posts**		
force (N)	deflection (μm)	gradient	force (N)	deflection (μm)	gradient
268.8	139.9	1.92	35.1	219.4	0.16
237.8	159.9	1.49	32.6	250.8	0.13
249.0	119.9	2.08	41.9	261.9	0.16
140.5	119.9	2.01	33.4	175.8	0.19
250.3	119.9	2.09	42.7	355.8	0.12
221.1	105.3	2.10	35.8	170.5	0.21
246.0	129.9	1.89	36.1	190.0	0.19
204.8	99.9	2.05	34.9	174.5	0.20
264.8	119.9	2.21	39.4	187.6	0.21
287.8	159.9	1.80	33.1	143.9	0.23
mean±SD	mean±SD	mean±SD	mean±SD	mean±SD	mean±SD
247.1±23.7	127.4±20.4	1.96±02	36.5±3.6	213.0±62.2	0.18±0.03

Student's *t* test revealed that ceramic posts were more rigid than stainless steel posts ($p<0.001$).

Visual examination of the ceramic posts revealed that in Group 4 (glass ionomer) all of the posts were clean after the retention test, indicating that the mode of failure was adhesive at the interface of the glass-ionomer cement and the posts. In Groups 2 and 3 traces of resin cement were found adhering to all of the posts, suggesting a partly cohesive mode of failure.

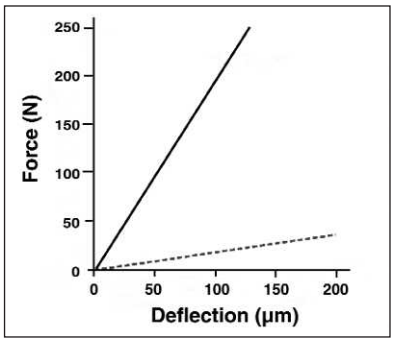


Figure 3. *Plots of the mean force versus deflection for the ceramic posts (solid line) and the stainless steel posts (dashed line).*

Electron Microscopy

Electron microscopic examination of the ceramic posts, as supplied by the manufacturer, revealed a rough, granular surface texture with no pattern (Figure 4A). Following sandblasting for 10 seconds with 50 μm alumina, the surface assumed a more regular pattern with parallel, circumferential corrugations approximately 4 μm in width (Figure 4B).

The fractured surfaces of the posts used in the bending tests were all of similar appearance (Figure 5). The portion of the post in compression, namely the half of the post adjacent to the moving head of the testing machine, suffered a fracture at right angles to its long axis, leaving a rough surface. From about midway through the post, the path of fracture deviated, turning 90 degrees to run along the long axis of the post, then turned abruptly again to exit the surface in tension at an oblique angle. The fractured surface of the portion in tension was smoother than that in compression.

DISCUSSION

As predicted from the results of other studies (Christel & others, 1989; Shimizu & others, 1993), the rigidity of the ceramic posts was very high. Ichikawa and others (1992) demonstrated that zirconia has twice the rigidity of polycrystalline aluminous ceramic. In the present study the zirconia posts exhibited higher resistance to bending than stainless steel, and also the typically brittle behavior of all ceramics, that of low ability to tolerate strain before fracture.

The high retention value of the serrated stainless-steel posts and the relatively low retention of the ceramic posts with glass-ionomer cement is consistent with previous work on the effects of shape and surface texture (Standlee, Caputo & Hanson, 1978). High retention can be predicted for parallel, serrated posts, and low retention can be predicted for tapered, smooth posts. Jørgensen and Holst (1967) demonstrated that cements with higher compressive strength produce a proportionally greater retention. This would partly explain the stronger retention of the ceramic posts with resin cement than with glass-ionomer cement. The silane coupling agents may have enhanced the bond between resin and post, as these materials are proven mediators of adhesion between resins and some ceramics (Eames & others, 1977). This may account for the retention of fragments of cement on ceramic posts after retention testing. However, the durability of bonds between silanated zirconia ceramic and BIS-GMA resin has been questioned by Kern and Wegner (1998), who found that the bond failed spontaneously after 150 days of water storage with thermocycling.

Sandblasting the ceramic posts could be predicted to enhance retention by improving mechanical interlocking of the cement and post. The regular pattern of grooves produced by the sandblasting reflected the way in which the posts were rotated in the stream of particles. The sandblasted group showed a trend towards improved retention. However, the retention of a number of specimens in this group was strong enough to result in fracture of the posts before the retention failed. The sandblasting process could have weakened the ceramic by introducing surface flaws that acted as crack initiation sites, or alternatively, the energy imparted by sandblasting may have modified the crystalline structure of the surface. A third possibility is

Table 2: *Comparison of the Mean Force (N) to Cause Retentive Failure of Group* with Other Groups*

Group	Force (N)	*t* test	Mann-Whitney U
1*	394±23		
2	190±95	<0.001	<0.001
3	118±21	<0.001	<0.001
4	54±90	<0.001	<0.001
2*	190±95		
3	118±21	>0.05	>0.1
4	54±90	<0.001	<0.001
3*	118±21		
4	54±90	<0.001	<0.001

Group 1: stainless steel post, resin cement
Group 2: sandblasted ceramic post, resin cement
Group 3: ceramic post, silane, resin cement
Group 4: ceramic post, glass ionomer cement

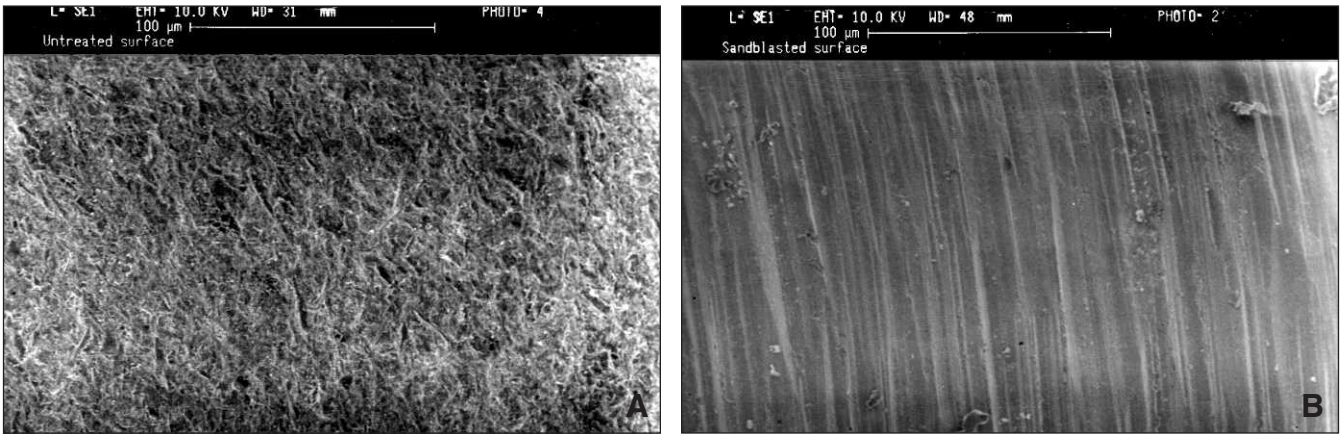


Figure 4. *SEM photographs of the ceramic posts. A: The surface of the ceramic post as supplied by the manufacturer. B: The surface of the ceramic post after sandblasting.*

Figure 5. *SEM photograph of a fractured ceramic post.*

simply that the ceramic was not weakened, but its tensile strength was exceeded during the retention test.

CONCLUSIONS

The ceramic root-canal posts (Cerapost) were significantly more rigid than the stainless-steel posts (Parapost).

The serrated, parallel, stainless-steel posts were significantly more retentive in roots than the smooth, tapering, ceramic posts.

A silane coupling agent and resin cement produced significantly stronger retention of the ceramic posts than that produced by glass-ionomer cement.

Sandblasting of the ceramic posts produced variable results and needs further investigation before it can be recommended.

(Received 4 January 1999)

References

Anusavice KJ ed (1996) *Phillips' Science of Dental Materials*, 10th edition, Philadelphia: WB Saunders pp 590-596.

Christel P, Meunier A, Heller M, Torre JP & Peille CN (1989) Mechanical properties and short-term *in-vivo* evaluation of yttrium-oxide-partially-stabilized zirconia *Journal of Biomedical Materials Research* **23(1)** 45-61.

Eames WB, Rogers LB, Feller PR & Price WR (1977) Bonding agents for repairing porcelain and gold: an evaluation *Operative Dentistry* **2(3)** 118-124.

Goerig AC, Michelich RJ & Schultz HH (1982) Instrumentation of root canals in molar using the step-down technique *Journal of Endodontics* **8(12)** 550-554.

Ichikawa Y, Akagawa Y, Nikai H & Tsuru H (1992) Tissue compatibility and stability of a new zirconia ceramic *in vivo Journal of Prosthetic Dentistry* **68(2)** 322-326.

Jørgensen KD & Holst K (1967) The relationship between the retention of cemented veneer crowns and the crushing strength of the cements *Acta Odontologica Scandinavica* **25(4)** 355-359.

Kern M, Simon MH & Strub JR (1998) Erste klinische Erfahrungen mit Wurzelstiften aus Zirkonoxidkeramik *Deutsche Zahnärztliche Zeitschrift* **53** 266-268.

Kern M & Wegner SM (1998) Bonding to zirconia ceramic: adhesion methods and their durability *Dental Materials* **14(1)** 64-71.

Schwartz RS, Summitt JB & Robbins JW (1996) Fundamentals of Operative Dentistry. Contemporary Concepts Chicago: *Quintessence* p 327.

Shimizu K, Oka M, Kumar P, Kotoura Y, Yamamuro T, Makinouchi K & Nakamura T (1993) Time-dependent changes in the mechanical properties of zirconia ceramic *Journal of Biomedical Materials Research* **27(6)** 729-734.

Standlee JP, Caputo A & Hanson EC (1978) Retention of endodontic dowels: effects of cement, dowel length, diameter, and design *Journal of Prosthetic Dentistry* **39(4)** 400-405.

Torbjörner A, Karlsson S & Ödman PA (1995) Survival rate and failure characteristics for two post designs *Journal of Prosthetic Dentistry* **73(5)** 439-444

©Operative Dentistry, 2000, **25,** 228-233

Marginal Hybrid Layer in Class V Restorations

C Prati • DH Pashley
S Chersoni • R Mongiorgi

Cinical Relevance

The quality of the hybrid layer created in dentin at the external margin of resin composite restorations (termed the marginal hybrid layer) may determine the longevity of such restorations, for it is very thin and exhibits porosites and grooves, indicating that it is not completely infiltrated with resin.

SUMMARY

The aim of this study was to evaluate the morphology of the hybrid layer (identified as marginal hybrid layer) along the cervical margins of Class V restorations using several bonding systems. Class V restorations were prepared *in vitro* at the CEJ in extracted third molars. Three different bonding systems were selected: Scotchbond 1 (Single Bond), Scotchbond MP, and Clearfil Liner Bond 2V. After finishing each restoration with disks, each margin was polished for one minute with polishing paste. The margins of half of the restorations were then treated with 10% phosphoric acid for five seconds, washed in deionized water, and then stored in water for 24 hours before SEM analysis. The margins of the other half of the restorations were treated with 10% phosphoric acid for five seconds, then with 1.5% NaOCl gel for two minutes to remove noninfiltrated collagen, then washed and stored in water for 24 hours. Each sample was gold coated and observed under SEM. A one-way ANOVA was performed to determine if there were any statistically significant differences in hybrid layer thicknesses. The thickness of the marginal hybrid layer measured under SEM was 1.5-2.5 µm thick in Scotchbond MP and Scotchbond 1 but varied from 0.0 to 12.0 µm in Clearfil Liner Bond 2V when observed after NaOCl postpolishing procedures. Far more porosities were seen in the marginal hybrid layer of Clearfil Liner Bond 2V. Enamel margins were free from a hybrid layer in both groups. This study demonstrated that collagen fibrils are not completely infiltrated by adhesive resin at the CEJ level along the cavorsurface margin of the restorations. The presence of noninfiltrated collagen along the external margins may reduce the sealing capability of bonding systems, leaving many open spaces and gaps, which may jeopardize the durability of the bond over time.

University of Bologna, School of Dentistry, Department of General Dentistry, Via San Vitale 59 – 40125 Bologna – Italy

Carlo Prati MD, DDS, PhD, research clinician of Bologna University–School of Dentistry

David H Pashley, DMD, PhD, regent's professor, Medical College of Georgia School of Dentistry, Department of Oral Biology-Physiology, Augusta, GA, 30912-1129

Stefano Chersoni, DD, postgraduate dentist assistant

Romano Mongiorgi, CD, PhD, professor, University of Bologna, Department of GeoEnvironmental Sciences

INTRODUCTION

One of the variables that may affect the longevity of Class V resin composite restorations is the quality of

the bond at the dentinal cavosurface margins (Prati & Pashley, 1992; Prati & others, 1995). There is a zone just beneath the cementum that has few dentinal tubules and that seems to have a low permeability to adhesive resins even after acid etching (Titley & others, 1994; Cagidiaco & others, 1997). There is a risk that acidic conditioners may demineralize the zone deeper than subsequently applied resin monomers can infiltrate, producing a poor-quality hybrid layer at this critical interface. The external margins of resin composites in dentin should be as perfect as possible, both macroscopically and microscopically, since these regions are often subgingival. Bacterial plaque formation on these surfaces places them in an environment where the pH can fall to low values during glycolysis and where many bacterial enzymes are produced. If resin infiltration is not perfect, these enviromental challenges may jeopardize the durability of the bond.

Few studies have examined hybrid layer formation in cementum or peripheral dentin. Tay and others (1994) probed the complexity of resin-sealed hard-tissue interfaces. They demonstrated that hybrid layers could be formed in cementum as well as dentin, but they did not challenge these hybrid layers with acids or NaOCl. Studies by Prati and others (1998) indicated that few resin tags are formed in peripheral dentin near the external margins of restorations. They speculated that the lack of dentinal tubules and their lateral branches may interfere with resin bonding to these areas, and that this may be the reason for the high incidence of marginal leakage in these sites that have so often been reported in the literature.

The purpose of this study was to test the null hypothesis that there is no difference in the thickness of hybrid layers formed at the dentinal cavosurface margins of Class V restorations from those formed more centrally using conventional vs self-etching bonding systems.

METHODS AND MATERIALS

Sample Preparation

Thirty freshly extracted third molars were obtained from young patients (mean age 23.9 years) and stored at 4°C in saline solution for no more than one month. Class V nonretentive cavities were prepared (3 mm in diameter x 2 mm deep) just below the cemento-enamel junction (CEJ) on the buccal surface, thereby creating a large dentin interface, which would permit SEM examination of resin-dentin bonds made under clinically relevant conditions.

In these cavity preparations, about half of the margins were in enamel, with the rest being in dentin. Medium and fine-grit diamond burs (Intensive, Lugano, Switzerland) were used in a high-speed water-cooled handpiece. All materials were placed according to the manufacturer's directions at room temperature (20-21°C) and at a relatively constant (50-62%) humidity. Table 1 lists the restorative materials. Dentin bonding agents were applied with small brushes. Resin composites were placed with a single increment using a stainless-steel spatula. The bonding agents were photocured with a light that delivered more than 400 mW/cm^2 (Visilux Command 2; 3M Dental Products, St Paul, MN 55144) for 20 seconds. Composite resins were then photocured for 60 seconds with the direction of the tip parallel to the composite surface.

Each restoration was immediately polished along the margins with wet silicon carbide papers (#600, #800, #1000, and #1200), then with diamond paste and polishing compounds, and stored in water at room temperature for 20 minutes, before gently washing with deionized water. All the samples were then exposed to 10% phosphoric acid gel (Bisco, Inc, Schaumburg, IL 60193) for five seconds and washed with deionized water. These techniques removed any debris produced during the polishing procedure. One-half of the samples (selected at random) were then exposed to a 1.5% NaOCl gel (Procter & Gamble, Cincinnati, OH 45202) for two minutes. After this time, each sample was copiously washed (for approximately two minutes) under tap water and deionized water. These procedures removed the superficial debris produced during the polishing procedures that could obscure the porosities and fractures along the margin of the restoration, and dissolved any collagen fibrils that were not enveloped by resin.

Each tooth was then immersed in 2% glutaraldehyde (pH 7.4) for 24 hours. Each sample was then dried at room temperature, gold coated, and inspected under SEM (Model 5400; JEOL, Tokyo, Japan).

SEM Examination of the Resin-Dentin Interface

Each cervical margin of restoration was inspected under SEM from one side of the CEJ to the other side. The thickness of the marginal hybrid layer was measured along the entire length every 100 microns. All the values were averaged to calculate the mean marginal dentin hybrid layer thickness for the inspected sample. In several restorations, the marginal hybrid layer was

Table 1: *Materials selected for the present study.*

DBA/Composite	Company
Clearfil Liner Bond 2V/Clearfil PhotoPosterior	Kuraray, Osaka, Japan
Scotchbond MP Plus/Z100	3M Dental Products, St. Paul, MN 55144
Scotchbond 1*/Z 100 (Scotchbond SingleBond* in USA)	3m Dental Products, St. Paul, MN 55144

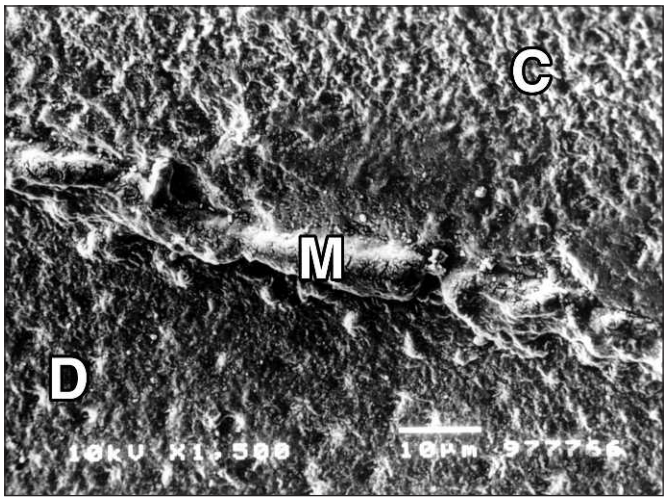


Figure 1. *SEM photograph of polished margin after acid treatment. No dentinal tubules are visible. C=composite; D=dentin; M=marginal hybrid layer. The interface and the marginal hybrid layer are free from porosites, but are not well defined. No porosites, discrepancies, and fractures are visible. Collagen is collapsed and probably filled all the voids.*

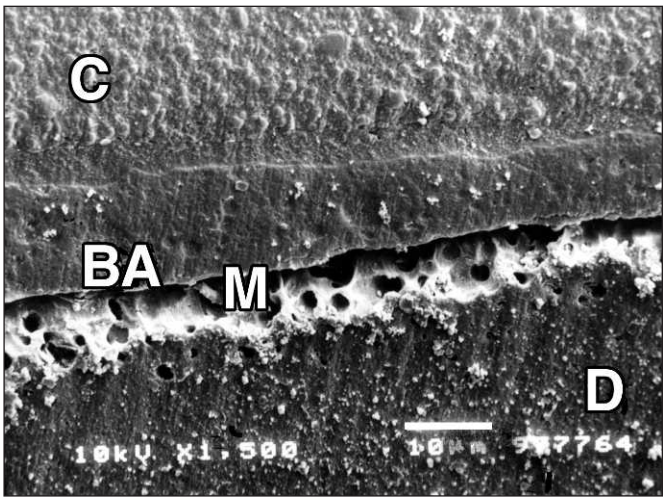


Figure 2. *SEM photograph of polished margin after acid treatment followed by NaOCl treatment. The dentin margin is rich in porosites and voids that probably represent the orifices of dentinal tubules and lateral branches. Composite (C). The layer of bonding agent (BA) and the layer of porosites correspond to the marginal hybrid layer (M) after the removal of non-resin infiltrated collagen. Dentin (D) is covered by a thin smear layer.*

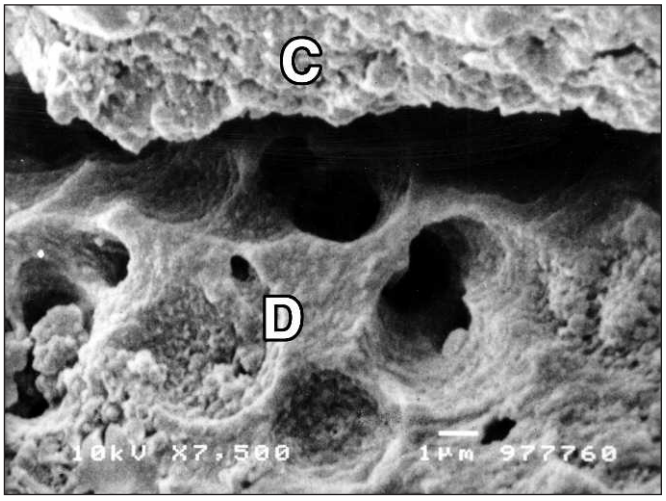


Figure 3. *SEM photograph of polished margin after acid treatment followed by NaOCl treatment. Higher magnification. Several channels of different dimensions (1.5 µm–0.5µm) are visible and partially covered by debris, probably smear layer produced during the polishing step. Composite (C) and dentin (D).*

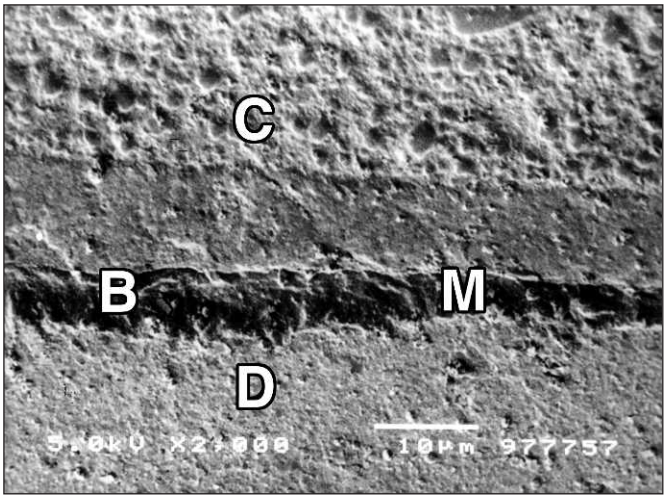


Figure 4. *SEM photograph of polished margin after acid treatment followed by NaOCl treatment. In this sample, the marginal hybrid layer (M) was completely sealed. Composite (C), bonding resin layer (B), and dentin (D) are well detectable. The thickness of marginal hybrid layer (M) is extremely thin (3-8 µm). No dentinal tubules are visible on the top of the dentin surface. Porosites and voids are extremely rare in this marginal hybrid layer.*

absent and a marginal gap and/or many porosities were observed along the margin. These alterations were visible close to the CEJ. In this case no measurements were done.

Statistical Analysis

The means and standard deviations of marginal hybrid layer thickness were calculated by averaging the individual values of each sample evaluated under SEM. A one-way ANOVA was performed to determine if there were any statistically significant differences in hybrid layer thicknesses. Post hoc multiple comparisons were made with Tukey's test using SigmaStat (SPSS, Chicago, IL 60302). Statistical analysis was defined as $p<0.05$.

RESULTS

Generally, there were few gaps seen between tooth structure and the resin composites, even though they were filled in a single increment. Scanning electron microscopic examination of the marginal hybrid layers revealed a number of important differences among the

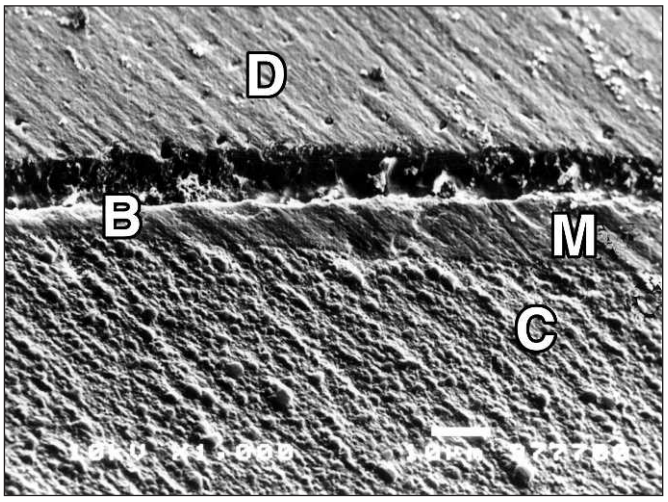


Figure 5. *SEM photograph of polished margin after acid treatment followed by NaOCl treatment. Marginal hybrid layer (M) was completely sealed. NaOCl treatment removed the not-infiltrated collagen along the marginal hybrid layer, leaving a groove or depression in place of the original flat surface. Composite (C), bonding resin layer (B), and dentin (D) with several open dentinal tubules are well detectable. The thickness of marginal hybrid layer (M) is extremely thin (3-8 µm).*

treatments. The micromorphology of these marginal hybrid layers depended upon whether they were acid etched only, or whether they were both acid etched and then treated with NaOCl. When the margins of the restorations were only acid etched to remove polishing debris, the resin-dentin interface appeared to be uniform, continuous, and free of large defects. The thickness of the marginal hybrid layer following acid etching appeared to be between 0.5-1.0 µm along the surface (Figure 1). There were no fractures or cracks seen along the resin-dentin interfaces, although these were most commonly seen in the resin-enamel interface. No marginal hybrid layer could be detected in resin-enamel interfaces using any of the bonding systems.

In contrast, when acid-etched specimens were subsequently treated with NaOCl, a number of porosities and voids were detected in the marginal hybrid layers in dentin (Figures 2 and 3). In some areas, NaOCl treatment removed the dentin (noninfiltrated collagen), leaving a groove or depression in place of the original flat surface of the marginal hybrid layer (Figures 4 and 5). This loss of tooth structure following NaOCl treatment was not seen in resin-enamel interfaces.

No cementum was noted on any of the specimens, because it was removed during the polishing procedures.

The external marginal hybrid layers produced by Scotchbond Multi-Purpose Plus and Single Bond following both acid and NaOCl treatments were about 2.0 to 2.5 µm thick (Figures 1 and 2), while those produced by Clearfil Liner Bond 2V were frequently thinner (1.2 µm). Not only were the marginal hybrid layers produced by Clearfil Liner Bond 2V thinner than those produced using conventional acidic conditioners, they were not uniform in thickness, with some areas being extremely thin, while others were relatively thick (Figure 4). When these hybrid layer thicknesses were compared to those made by the same materials on internal superficial dentin (Table 2), it was clear that those made by the use of conventional 35% phosphoric acid conditioner were much thicker ($p<0.05$). They were also thicker using the self-etching system, although the difference did not reach statistical significance (Table 2).

DISCUSSION

The bond created by the adhesive agents and the dentin was apparently strong enough to resist the forces of polymerization contraction (Davidson, de Gee & Feilzer, 1984). The hybrid layers created in dentin at the external margin of the root were thinner than those that we have reported in more central dentin using the same products (Table 2). This is thought to be due to differences both in the number of tubules/mm^2 (Yoshiyama & others, 1995) and in their orientation (Schüpbach, Krejci & Lutz, 1997). For instance, Prati and others (1998) reported dentin hybrid layer thicknesses of 3 µm using Scotchbond Multi-Purpose Plus and 2 µm using Single Bond (Scotchbond 1 in Europe) in superficial central dentin. Goracci, Mori, and Bazzucchi (1995) reported hybrid layers of 3-5 µm thick in specimens bonded *in vivo*. Nakajima and others (1995) reported hybrid layers of 2.3 ± 0.4 µm thick in normal dentin when using Scotchbond Single Bond. Using Clearfil Liner Bond 2V, Nakajima and others (1995) obtained hybrid layers in normal superficial central dentin that were only 0.5 µm thick. Yoshiyama and others (1996) reported that the hybrid layers were thicker when the dentinal tubules were cut in cross section compared to when the bonded surface was parallel to the tubules. Using Clearfil Liner Bond 2 (the light-cured version of Liner Bond 2V), the hybrid layers were <0.5 µm thick when the primer was applied to cavity walls cut parallel to dentinal tubules, but were 1.3 µm thick when applied to dentinal tubules cut in cross section. The lack of tubules in this region may hinder resin infiltration during bonding.

Similar results were reported by Schüpbach and others (1997), who obtained thinner hybrid layers on dentin with tubules prepared parallel to the dentin surface than when they were cut in cross section. It is also possible that this region of external dentin is more acid resistant than deeper areas.

However, there was no correlation between the thickness of hybrid layers and bond strength (Finger, Inoue & Asmussen, 1994; Nakajima & others, 1995; Yoshiyama & others, 1996). It is important that whatever hybrid layer is formed must be of high quality. If acidic conditioners etch more deeply into dentin than

Table 2: *Thickness of marginal (external) hybrid layers measured after NaOCl post-polishing treatments.*

DBA/Composite	Hybrid Layer (µm ± S.D.; N=10			
	External Marginal HL		Internal HL*	
	Mean ± SD	Range	Mean ± SD	Range
Clearfil Liner Bond 2V/Clearfil	1.2 ± 1.5	00.00-12.00	1.5 ± 0.5	1.0 - 2.0
Scotchbond MP Plus/Z100	2.0 ± 1.2 a	0.5 -3.0	3.0 ± 1.0	2.0 - 4.5
Scotchbond 1*/Z 100	1.8 ± 0.5 a	0.0 - 2.5	2.0 ± 1.0	1.5 - 2.5

Groups connected by the same letter are not significantly different ($p<0.05$)
•Values from Prati et al., *Operative Dentistry* 1998; **23**; 185-194.

adhesive resins can follow, there will be naked collagen fibrils at the bottom of the hybrid layer that may hydrolyze over time. Self-etching primers seem to produce relatively high bond strengths with relatively thin hybrid layers (Nakajima & others, 1995; Yoshiyama & others, 1996), but those formed at the dentin margin in this study were not uniform and were occasionally absent, thereby producing marginal gaps. It appears that this region of resin restorations needs to be carefully studied. Dentin in this region of the root, immediately below cementum, has a relatively low number of dentinal tubules that have an orientation parallel to the cavity wall (Garberoglio & Brännstrom, 1976; Cagidiaco & others, 1997). Cagidiaco and others (1997) reported that the number of dentinal tubules/mm^2 decreased as the dentin of approximal boxes of Class II cavity preparations progressed more peripherally. In fact, they reported that dentin just beneath the cementum was devoid of tubules. This confirms earlier TEM studies by Furseth (1974), who could not identify a true boundary between cementum and dentin, and who noted the absence of tubules in the most peripheral 100 mm of dentin. A recent proton-probe study of the distribution of fluoride across normal root dentin showed very high fluoride concentrations not only in cementum but in the dentin just beneath the cementum (Shu & others, 1998). Even higher values were found in root dentin at the edge of early carious lesions. These high fluoride levels may interfere with the ability of acidic conditioners to remove the mineral crystallites from collagen fibrils in these areas and may be responsible, in part, for the presence of nonuniform etching and nonuniform resin infiltration in this critical area. Ciucchi (1997), using a microtensile bond testing technique, obtained relatively lower bond strengths of Scotchbond Multi-Purpose to the outermost region of approximal boxes prepared into dentin just beneath the CEJ, than in more central regions. This marginal hybrid layer should also be examined for nanoleakage (Sano & others, 1994, 1995) to determine how far into the dentin the voids and porosities extend.

NaOCl postpolishing procedures removed only the debris from the marginal hybrid layer and permitted the evaluation of all the porosities and alterations created during the application of bonding systems and during the shrinkage of composite materials. Cavosurface margins in dentin provide an open door for marginal leakage toward the pulp and must be closed with a sealing hybrid layer. In practice, a perfect hybrid layer may seal this area and stop any microleakage and increase the longevity of such restorations. On the contrary, an irregular and porous marginal hybrid layer may increase secondary caries and dentin sensitivity. Future investigations are needed to evaluate the influence of acid-cariogenic solutions and saliva proteolitic enzymes on the morphology of the marginal hybrid layer (Prati & others, 1999).

CONCLUSIONS

1. Longer etching times and an application of a second layer of bonding agent after finishing aided marginal sealing of cervical margins of Class V restorations that involved dentin.

2. The use of NaOCl on cavity preparations after acid etching produced porosity and voids in the marginal hybrid layer and removed dentin tooth structure in some teeth.

Acknowledgment

The authors would like to thank Shirley Johnston for secretarial support. This work was supported, in part, by grant DE 06427 from the National Institute of Dental and Craniofacial Research.

(Received 5 January 1999)

References

Cagidiaco MC, Ferrari M, Vichi A & Davidson CL (1997) Mapping of tubule and intertubule surface areas available for bonding in Class V and Class II preparations *Journal of Dentistry* **25(5)** 379-389.

Ciucchi B (1997) *Bonding Characteristics of a Resin Composite to Dentine Walls in a Class II Cavity, in vitro* Thése d'Habilitation, Geneva, Switzerland: University of Geneva.

Davidson CL, de Gee AJ & Feilzer A (1984) The competition between the composite-dentin bond strength and the polymezization contraction stress *Journal of Dental Research* **63(12)** 1396-1399.

Finger WJ, Inoue M & Aamussen E (1994) Effect of wettability of adhesive resins on bonding to dentin *American Journal of Dentistry* **7(1)** 35-38.

Furseth R (1974) The structure of peripheral root dentin in young human premolars *Scandinavian Journal of Dental Research* **82(8)** 557-561.

Garberoglio R & Brannstrom M (1976) Scanning electron microscopic investigation on human dentin tubules *Archives of Oral Biology* **21(6)** 355-362.

Goracci G, Mori G & Bazzucchi M (1995) Marginal seal and biocompatibility of a fourth-generation bonding agent *Dental Materials* **11(6)** 343- 347.

Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama M, Ebisu S, Ciucchi B, Russell CM & Pashley DH (1995) Tensile bond strength and SEM evaluation of caries-affected dentin using dentin adhesives *Journal of Dental Research* **74(10)** 1679-1688.

Prati C, Chersoni S, Mongiorgi R & Pashley DH (1998) Resin-infiltrated dentin layer formation of new bonding systems *Operative Dentistry* **23(4)** 185-194.

Prati C, Chersoni S, Pistani A, Montanari G & Mongiorgi R (1999) Effect of caries solution on marginal morphology of dentin-composite interfaces *Journal of Dental Research* **78** Abstracts of Papers p 477 Abstract 2976.

Prati C, Ferrieri P, Galloni C, Mongiorgi R & Davidson CL (1995) Dentine permeability and bond quality as affected by new bonding systems *Journal of Dentistry* **23(4)** 217-226.

Prati C & Pashley DH (1992) Dentin wetness, permeability and thickness and bond strength of adhesive systems *American Journal of Dentistry* **5(1)** 33-38.

Sano H, Takatsu T, Ciucchi B, Horner JA, Matthews WG & Pashley DH (1995) Nanoleakage: Leakage within the hybrid layer *Operative Dentistry* **20(1)** 18-25.

Sano H, Yoshiyama M, Ebisu S, Burrow MF, Takatsu T, Ciucchi B, Carvalho R & Pashley DH (1995) Comparative SEM and TEM observations of nanoleakage within the hybrid layer *Operative Dentistry* **20(4)** 160-167.

Schüpbach P, Krejci I & Lutz F (1997) Dentin bonding: effect of tubule orientation on hybrid-layer formation *European Journal of Oral Science* **105(4)** 344-352.

Shu M, Pearce EI, Sissons CH, Coote GE & Miller JH (1998) Fluoride distribution in sound and carious root tissues of human teeth *Caries Research* **32(4)** 239-246.

Tay FR, Gwinnett AJ, Pang KM & Wei SHY (1994) Structural evidence of a sealed tissue interface with a total-etch wet-bonding technique *in vivo Journal of Dental Research* **73(3)** 629-636.

Titley K, Chernecky R, Maric B & Smith D (1994) Penetration of a dentin bonding agent into dentin *American Journal of Dentistry* **7(4)** 190-194.

Yoshiyama M, Carvalho R, Sano H, Horner J, Brewer PD & Pashley DH (1995) Interfacial morphology and strength of bonds made to superficial versus deep dentin *American Journal of Dentistry* **8(6)** 297-302.

Yoshiyama M, Sano H, Ebisu S, Tagami J, Ciucchi B, Carvalho RM, Johnson MH & Pashley DH (1996) Regional strengths of bonding agents to cervical sclerotic root dentin *Journal of Dental Research* **75(6)** 1404-1413.

©*Operative Dentistry, 2000,* **25,** 234-236

Clinical Techniques/Case Reports

Techniques in Recording Centric Relation

DH Hartzell • AJ Maskeroni • FC Certosimo

PURPOSE

A variety of different clinical techniques are currently being used to obtain centric relation (CR) records. The techniques can be categorized by those in which the operator guides the mandible and those made by the patient's own muscular action. Previous investigations have indicated a condylar position to be superior in the glenoid fossa when the patient's own muscles are relied on for the placement (Williamson, 1977; Williamson, 1978). It has been suggested that of the three planes of space for condylar position, superior is the most important. The article reviews the techniques that best accomplish the uppermost position of the condyle.

BACKGROUND

CR is a classic reference and treatment position. Such a position provides restorative dentists with a repeatable and stable position from which to work when reconstructing the dentition. Much controversy exists regarding the concept of CR. Early gnathologists (McCollum, 1939; Posselt, 1968) described the condyle as being at the rearmost and uppermost position in their respective fossae when fully seated in CR. Dawson (1989) has pointed out that "rearmost" and "uppermost" are misleading because the two positions are not compatible. He noted that the condyles are not in their rearmost position when they are in the uppermost position and vice versa. Centric relation is presently defined by Roth (1981), Williamson (1981), and Dawson (1989) as the relationship of the mandible to the maxilla when properly aligned condyles and discs are in their most superior position in the fossae and braced against the eminentia, irrespective of tooth position or vertical

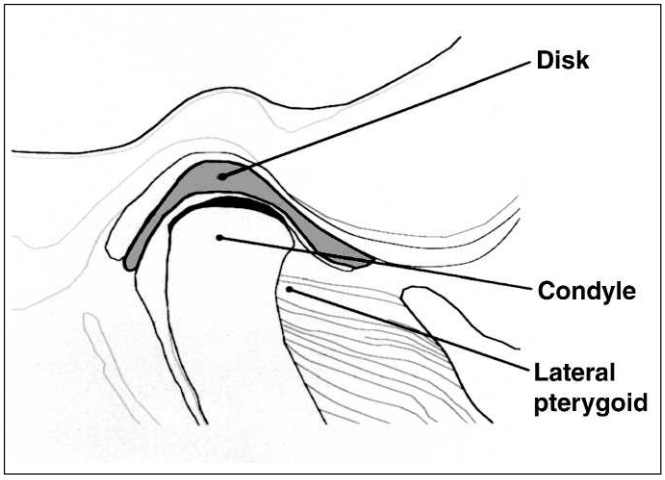


Figure 1. *Anatomic centric relation.*

Advanced Clinical Program and Advanced Education in General Dentistry, Naval Dental Center, Mid-Atlantic Region, Norfolk, VA 23511.

David H Hartzell, DDS, LCDR, USN, assistant director

Alfred J Maskeroni, DDS, CAPT, USN, director of orthodontics, Naval Post-Graduate Dental School, 8901 Wisconsin Ave, Bethesda, MD 20889

Fred C Certosimo, DDS, associate professor, Medical College of Virginia, School of Dentistry, 520 N 12th St, PO Box 980566, Richmond, VA 23298

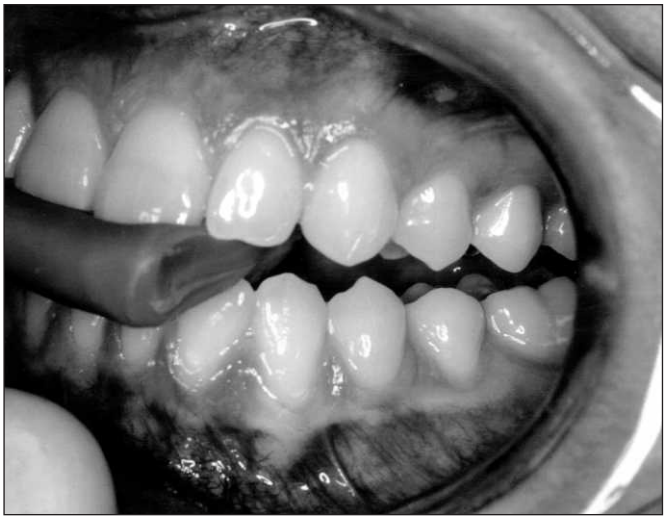


Figure 2.

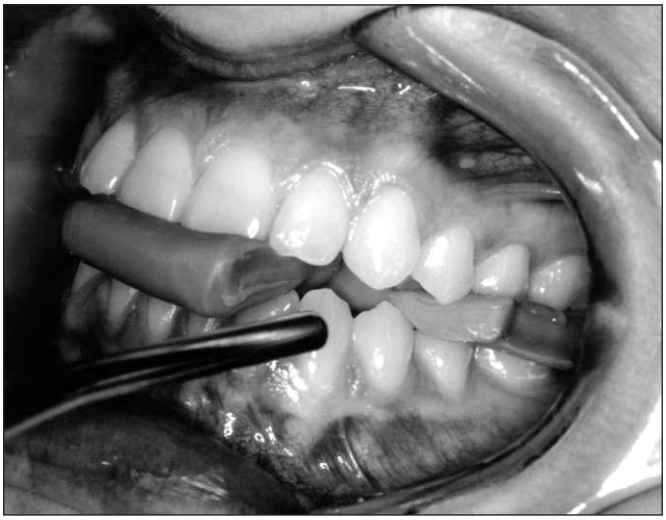


Figure 3. *Anterior stop with posterior wax.*

dimension. (Figure 1) Moffett (1978) suggested that this position is physiologically described and is an acceptable reference position for treatment.

TECHNIQUES

There are several clinical techniques, however, that emphasize superior positioning of the condyles. The anterior jig advocated by Lucia (1958) and the leaf gauge advocated by Long (1973) are examples of anterior stops. Use of an anterior stop separates the posterior teeth, thereby, allowing neuromuscular deprogramming and eliminating possible tooth interferences that would otherwise guide the mandible into maximum intercuspation. The chin-point guidance technique for superior positioning was first described by McCollum (1927) and involves keeping the posterior teeth separated by downward chin-point pressure. It is believed that this allows the elevator muscles to seat the condyles into CR. The bilateral manipulation (Dawson, 1989) method employs a specific superior guidance to the mandible at the same time the operator applies downward pressure with the thumbs at the chin and upward pressure at the angle of the mandible, attempting to seat the condyles in the most superior position. This method has been shown to have a consistent reproducibility (Kantor, 1972; Hobo, 1985). Bilateral manipulation methods require a combination of delicacy and timing when manipulating the mandible so that muscles that protrude the mandible are not triggered to contract by applying pressure at the wrong time or in the wrong direction (Dawson, 1989). It is, therefore, technique sensitive. The muscle seated centric registration, using a two-piece wax bite, is a technique advocated by Roth (1981). This technique incorporates the benefits of both mandibular manipulation and an anterior stop to register the most superior anterior position of the condyles. The anterior stop (Figure 2) is fabricated in wax using downward chin-point guidance with upward pressure at the gonion to position the patient's condyles superiorly and anteriorly. The hardened anterior stop, once verified, is then used along with a softened posterior piece of wax (Figure 3) to seat the condyles in the most superior position using the patient's own musculature. Lundeen (1974) found that heavy muscular contraction by a patient with a rigid anterior stop seated the condyle in the most superior position in a consistent and reproducible manner. All centric registration techniques have limitations. This muscle-seated CR registration seems to be less technique sensitive. It allows the patient's own muscles to produce the best record of CR for that patient on that particular day. The clinician should be cognizant of patients with parafunctional activity of the masticatory and cervical muscles. Fatigued muscles may lead to pain, hypoactivity, and altered range of motion (Mao, 1993; Kroon, 1992).

SUMMARY

1. Muscle seated centric registration is a reproducible method of obtaining centric relation (Wood, 1994).

2. The muscle-seated CR record provides a consistent, accurate, less technique-sensitive CR record of condylar position.

The opinions or assertions contained in this article are those of the writers and are not to be construed as official or reflecting the views of the Department of the Navy, Department of Defense, or the US Government.

Note: The mention of any brand names in this article does not imply recommendation or endorsement by the Department of the Navy, Department of Defense, or the US Government.

(Received 19 November 1999)

References

Dawson PE (1989) Evaluation, diagnosis, and treatment of occlusal problems. St Louis: CV Mosby 28-55.

Hobo S & Iwata T (1985) Reproducibility of mandibular centricity in three dimensions *Journal of Prosthetic Dentistry* **53(5)** 649-654.

Kantor ME, Silverman, SI, Garfinkle L (1972) Centric relation recording techniques-A comparative investigation *Journal of Prosthetic Dentistry* **28(6)** 593-600.

Kroon GW and Naeije M (1992) Electromyographic evidence of local muscle fatigue in a subgroup of patients with myogenous craniomandibular disorders *Archives Oral Biology* **37(3)** 215-218.

Long JH (1973) Locating centric relation with a leaf gauge *Journal of Prosthetic Dentistry* **29(6)** 608-610.

Lucia VO (1958) A technique for recording centric relation *Journal of Prosthetic Dentistry* **14** 492-505.

Lundeen HC (1974) Centric relation records: The effect of muscle action *Journal of Prosthetic Dentistry* **31(3)** 245-251.

McCollum BB (1939) Fundamentals involved in prescribing restorative dental remedies *Dental Items of Interest* **61** 724, 852.

McCollum BB (1927) Function-factors that make mouth and teeth a vital organ *Journal of the American Dental Associatio*n **14** 1261-1271.

Mao J, Stein RB, Osborne JW (1993) Fatigue in Human Jaw Muscles : A review *Journal of Orofacial Pain* 1993 **7(2)** 135-142.

Moffett B (1978) A biologic consideration of centric relation based on skeletal and connective tissue responses In *Occlusion, a state of the art.* Celenza, FV & Nasedkin, JN eds Quintessence Publishing Co, Inc Chicago 13-18.

Posselt V (1968) *Physiology of Occlusion and Rehabilitation* ed 2 Oxford Blackwell.

Roth RH (1981) Functional Occlusion for the Orthodontist Part 1 *Journal of Clinical Orthodontics* **15(1)** 32-51.

Williamson EH (1977) The effect of bite plane use on terminal hinge axis location *Angle Orthodontics* **47(1)** 25-33.

Williamson EH (1978) Laminographic study of mandibular condyle position when recording centric relation *Journal of Prosthetic Dentistry* **39(5)** 561-564.

Williamson EH (1981) Occlusion and TMJ Dysfunction *Journal of Clinical Orthodontics* **15(5)** 333-410.

Wood DP (1994) Reproducibility of the centric relation bite registration technique *Angle Orthodontics* **64(3)** 211-217.

©Operative Dentistry, 2000, **25,** 237-238

Expedited Custom Post and Core Technique

SG Alfano • MW Tyler

Custom fabricated cast post and cores have several advantages. By fabricating a post that fits the canal space of a tooth, conservation of radicular tooth structure is better achieved. If the canal space is prepared to a sufficient length, the success of the custom post and core restoration is high (Morgano & Milot, 1993; Bergman & others, 1989). Custom post and cores may be cast with the alloy of choice. Precious metal may be used, or if increased strength is required, a base metal alloy may be used.

One disadvantage of cast post and core restorations is that more chair time is required to fabricate and cement as compared to a prefabricated post (Abou-Ross, 1992; Johnson & others, 1976). A technique of rapid burnout and casting has been presented (Compagni & Majchrowicz, 1991); however, considerable chair time is still lost. In addition, many dental offices are not equipped with a casting machine. The article presents a clinical technique that eliminates an appointment while fabricating a custom cast post and core restoration.

Technique

1. Prepare post space and fabricate acrylic post and core pattern.

2. Prepare the core portion. (Figure 1.)

Naval Post-Graduate Dental School, 8901 Wisconsin Ave, Bethesda, MD 20889

Stephen G Alfano, DDS, LCDR, USNR, 3rd-year resident, Department of Prosthodontics

Michael W Tyler, DDS, CAPT, USN, chairman, Department of Operative Dentistry

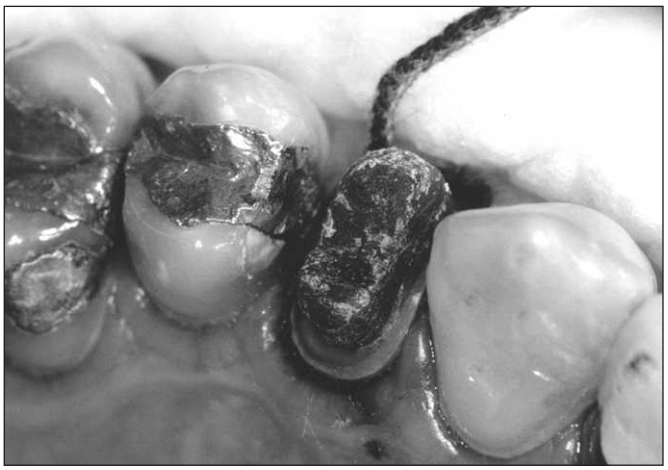


Figure 1. *The coronal portion of the acrylic pattern is prepared.*

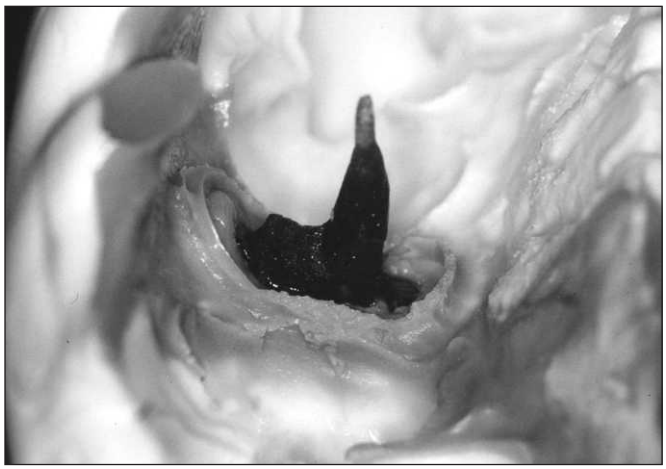


Figure 2. *The post and core pattern is removed from the tooth in the impression.*

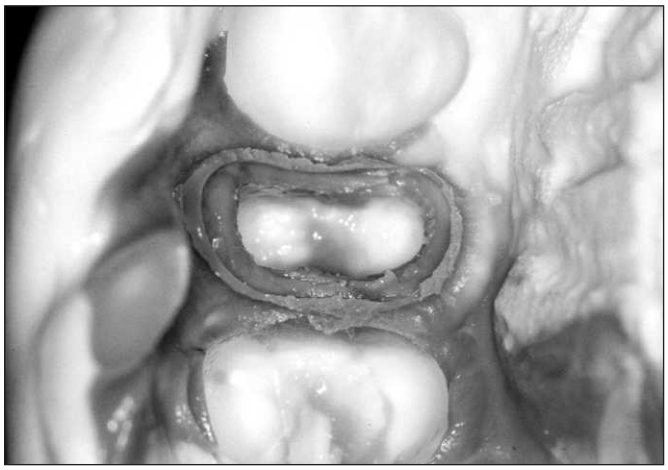


Figure 3. *The final impression after removing the post and core pattern.*

3. Retract tissue and make final impression. Care must be taken to ensure the post and core remain fully seated during the impression step. The post and core pattern will be removed from the tooth in the impression. (Figure 2.)

4. Remove the post and core from the impression. The impression will resemble an impression of a prepared tooth. (Figure 3.)

5. Send the post and core pattern and the impression to the laboratory to be fabricated.

6. The post and core can be cemented the same day as the crown. The post and core must be fully seated upon cementation in order for the crown to fit adequately.

The patient no longer has to return for cementation of the post and core and crown on separate days. The appointment time is reduced from three to two clinical appointments.

(Received 7 December 1999)

References

Abou-Rass M (1992) Post and core restoration of endodontically treated teeth C*urrent Opinion in Dentistry* **2** 99-107.

Bergman B, Lundquist P, Sjogren U, and Sundquist, G (1989) Restorative and endodontic results after treatment with cast post and cores *Journal of Prosthodontic Dentistry* **61(1)** 10-15.

Campagni WV and Majchrowicz M (1991) An accelerated technique for casting post and core restorations *Journal of Prosthodontic Dentistry* **66(2)** 155-156.

Morgano SM and Milot PM (1993) Clinical success of cast metal post and cores *Journal of Prosthodontic Dentistry* **70(1)** 11-16.

Johnson JK, Schwartz NL, and Blackwell RT (1976) Evaluation and restoration of endodontically-treated posterior teeth *Journal of the American Dental Association* **93(3)** 597-605.

©Operative Dentistry, 2000, **25,** 239-243

Conservative Anterior Tooth Replacement Using Fiber-Reinforced Composite

JC Meiers • MA Freilich

INTRODUCTION

Patients presenting with missing anterior teeth now have the option of having these teeth replaced with an adhesive, intracoronal inlay bridge that uses pre-impregnated, fiber-reinforced composite (FRC) technology. This fiber-reinforced composite serves as a framework that replaces the classic metal framework in porcelain fused-to-metal prosthesis. The FRC substructure is then layered with an advanced particulate composite, which would correspond to the porcelain layered over the metal framework. The combination of this two-phase polymer prosthesis combines the best characteristics of the fiber-reinforced composite (strength and rigidity) with those of the particulate composite (wear resistance and esthetics) (Fahl & Casellini, 1997; Freilich & others, 1998b).

Creating this fiber-reinforced composite bridge has been made possible by developing a manufacturing process that couples glass fibers into a resinous matrix, resulting in a uniformly impregnated framework (Goldberg & Burstone, 1992; Zanghellini, 1997) (Figure 1). This framework construction is different from other commercially available fiber-reinforcing materials, such as the plasma-treated woven polyethylene fibers in Ribbond (Ribbond™, Seattle, WA 98101) and Connect™ (Kerr, Orange, CA 92867) or the etched, silanted-glass fibers in GlasSpan™ (GlasSpan, Exton, PA 19341), which the clinician or lab technician needs to hand impregnate with either unfilled resin or a particulate composite. The preimpregnated FRC creates a substructure that has been shown to support two to three times the load and have 10 times the flexure modulus of the hand-impregnated designs (Freilich, Karmaker & Burstone, 1997; Goldberg & others, 1998). This preimpregnated FRC is relatively translucent and requires no masking, which allows for a relatively thin (approximately 0.5 mm) layer of particulate veneering composite to be placed over the FRC substructure to provide an esthetic appearance. This creates the ability to place supragingival margins, which can be made to easily blend in with the adjacent tooth structure on the abutment teeth. The lack of metal and opaque materials allow for a good translucency and a very lifelike and natural appearance of the prosthesis.

Department of Prosthodontics and Operative Dentistry, University of Connecticut School of Dental Medicine, 263 Farmington Ave, Farmington, CT 06030-1615

Jonathan C Meiers, DMD, MS, associate professor

Martin A Freilich, DDS, associate professor

CHARACTERISTICS OF COMMERCIALLY AVAILABLE PRE-IMPREGNATED FRC SYSTEMS

Two commercially preimpregnated FRC materials are available today: FibreKor™ (Jeneric/Pentron, Wallingford, CT 06492), with its veneering particulate composite, Sculpture, and Vectris™ (Ivoclar North America, Amherst, NY 14228), with its veneering particulate composite, Targis™. FibreKor is composed of parallel S-glass fibers made up of silica, magnesia, and alumina (Figure 2A), preimpregnated in BIS-GMA, polycarbonate dimethacryate, and ethoxylated A-dimethacrylate. Sculpture™ particulate composite is composed of polycarbonate dimethacrylate, ethoxylated A-dimethacrylate, and triethyleneglycol dimethacrylate plus barium glass and fumed silica as filler particles.

The Sculpture™ particulate composite veneering material has an average particle size of 0.6 μm, is 75 % filled by weight, and is light and heat cured. Vectris™ has three orientations of its R-glass fibers, which are imbedded in a BIS-GMA, decandiol dimethacrylate, and urethane

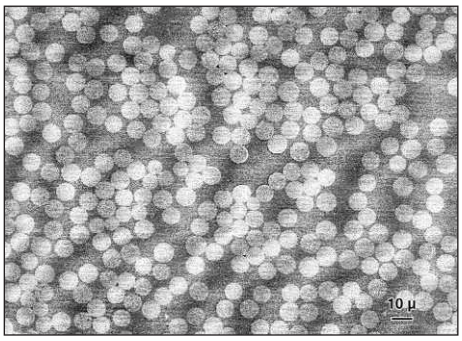


Figure 1. *SEM of cross sections of FibreKor™ samples showing glass fibers imbedded in resin. Original magnification X500.*

dimethacrylate: Vectris™ Pontic, which has parallel glass fibers similar to the FibreKor™ orientation; Vectris™ Frame (Figure 2B), which is composed of a satin weave of 30-degree bias-cut fibers; and Vectris Single, which is a plain satin-weave orientation. The Targis™ particulate composite veneering material is composed of the same basic resin matrix with the addition of barium glass, mixed oxides, and dispersed silica as fillers. Targis™ has an average particle size of 1 µm, is 78% filled by weight, and is light and heat cured. Currently, the manufacturers have recommended that Sculpture™/FibreKor™ be used for both anterior and posterior tooth replacement, while Targis™/Vectris™ is advocated only for posterior tooth replacement.

CASE SELECTION FOR A FIXED FRC PROSTHESIS

Indications for selection of a fiber-reinforced composite prosthesis include: (1) requiring an optimal esthetic result; (2) the desire for a metal-free, porcelain-free prosthesis; (3) the desire to decrease the potential wear of the opposing teeth (versus porcelain); and (4) the desire to use an adhesive approach to the abutment teeth (Freilich & others, 1998a).

Contraindications for selecting a fiber-reinforced composite prosthesis are: (1) the inability to obtain good moisture control, where the use of an adhesive technique cannot be successfully performed; (2) a prosthesis that involves two or more pontics, ie, long-span bridges; (3) patients who exhibit parafunctional habits; (4) the presence of unglazed porcelain opposing the prosthesis; and (5) patients who abuse alcoholic substances (Freilich & others, 1998a).

Using adhesive cementation techniques requires a contamination-free field. Rubber dam isolation is ideal when the situation allows its application. Margins should ideally be at or above the gingival margin, and if subginigival, the sulcus must be healthy and not showing signs of hemorrhage or exudate. Replacing more than one tooth with this type of prosthesis is not recommended due to lack of long-term documentation regarding this material's ability to support the tooth replacement in larger edentulous spans. The potential for increased wear or fracture exists if this type of prosthesis is placed in patients who brux. Patients who abuse alcohol may encounter increased surface degradation.

INLAY DESIGN FRC PROSTHESIS

The use of an inlay design for the retainers is an attractive, conservative alternative that can be used with the FRC prosthesis. The criteria for selecting abutment teeth that could be used successfully with this approach are: (1) teeth with minimal to no existing restorations; (2) abutment teeth that are acceptable in shape, position, and color; and (3) abutment teeth with enough buccolingual thickness to permit a 2 mm-deep intracoronal preparation.

Abutment teeth are prepared with a Class II inlay preparation design with a short incisogingival approximal step. The preparation design can incorporate an existing Class III restoration, if present. The depth and width of the lingual horizontal groove must be around 2 mm. The width of the approximal step is equal to the lingual extension, with its gingival extent determined by the CEJ and the free gingival margin. A length of 1.5 mm-2 mm with the gingival margin located coronal to the free gingival margin and CEJ would be ideal. There is no reason to prepare a full-length approximal box versus a step, because the FRC cannot be placed apical to the contact area to act as a connector and function as the substructure of the connector while maintaining adequate embrasure form. Only particulate composite would be used to fill the box apical to the contact/connector area, and this would not add any strength to the prosthesis. The gingival floor of the approximal box has a width of at least 1.5 mm. These dimensions are critical because it permits the lab technician to place enough bulk of FRC material for adequate strength, especially in the interproximal connector region. All margins should be butt joint and placed in enamel with the gingival margin located supragingivally.

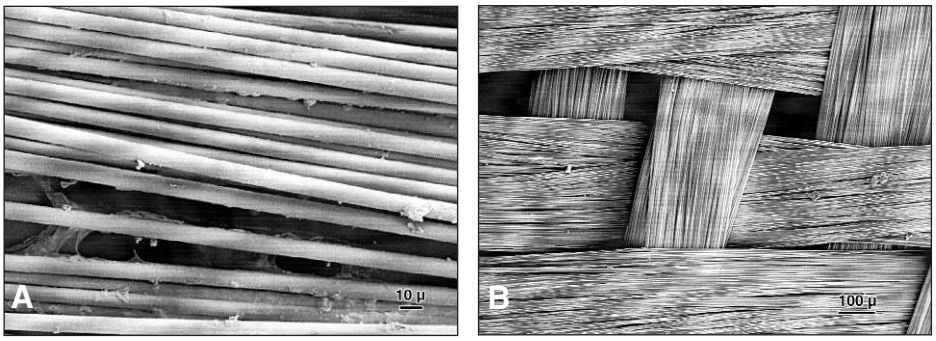


Figure 2A and 2B. *SEMs showing orientation of glass fibers after the resin has been removed. a) FibreKor™ FRC sample showing unidirectional orientation of glass fibers. Original magnification X500; b) Vectris™ Frame FRC showing its weave pattern. Original magnification X100.*

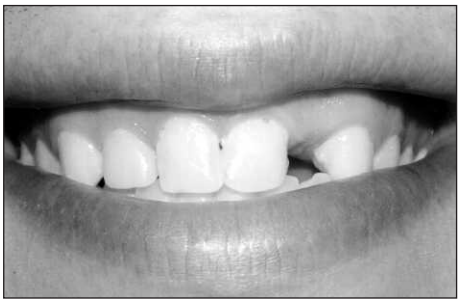


Figure 3. *Facial view of patient with missing lateral incisor. Short clinical crowns on canines and gingival height discrepancy on the central incisors.*

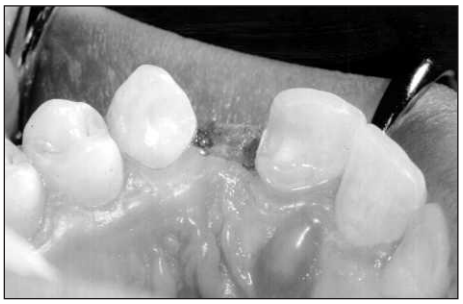


Figure 4. *Abutment teeth have been prepared. Electrosurgery was performed on proximal gingiva around abutment teeth to provide crown lengthening in order to provide the proper length for the proximal step and to keep the gingival margin supragingival.*

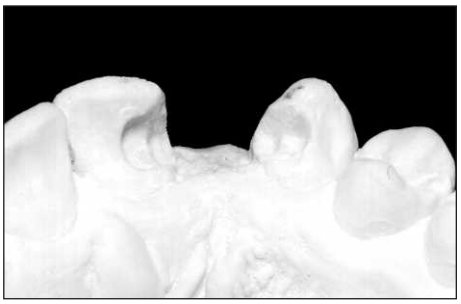


Figure 5. *Stone model showing details of inlay preparations on central incisor and canine. Preparations are kept supragingival. Lingual view.*

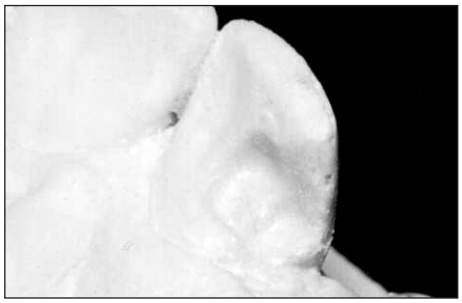


Figure 6. *Stone model showing lingual groove and proximal step on central incisor.*

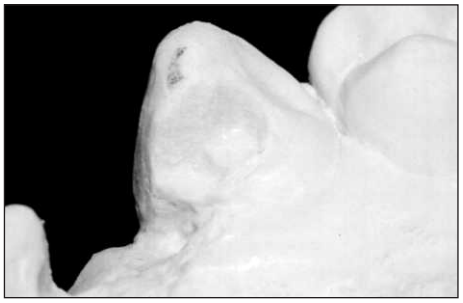


Figure 7. *Stone model showing lingual slot and proximal step on canine.*

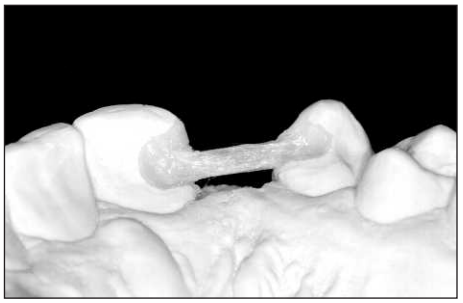


Figure 8. *Initial pontic formation with FRC. Particulate composite is laid first in the lingual groove and proximal step and then six layers of FRC strips are placed bridging the two proximal steps of the abutment teeth.*

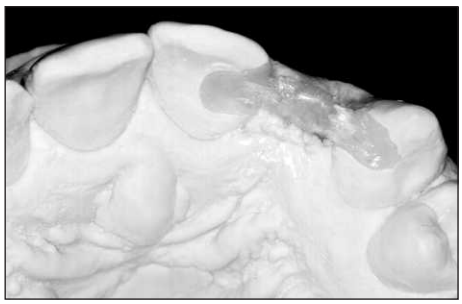


Figure 9. Lingual view of completed FRC substructure with developed pontic Shape. The FRC pontic substructure functions similarly to the metal substructure in PFM design in that it fully supports all aspects of the particulate composite overlay.

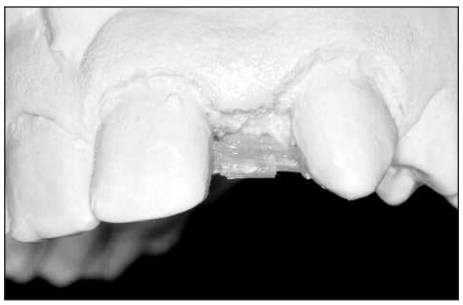


Figure 10. *Facial view of completed FRC substructure.*

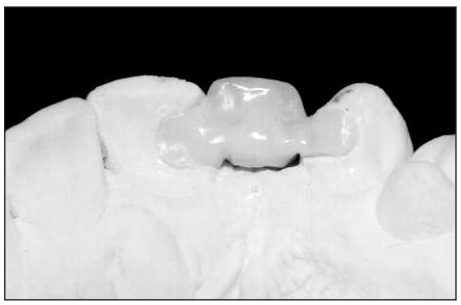


Figure 11. *Lingual view of completed FRC inlay bridge on stone model.*

CASE PRESENTATION OF AN INLAY DESIGN BRIDGE

This patient presented with a missing maxillary left central incisor from a failure of a previously placed adhesive chairside bridge, which had slot preparations as the pontic (Figure 3). The patient desired a conservative approach, which eliminated the use of conventional full-crown preparations. Two options were presented: an etched metal, resin-bonded approach or an inlay FRC approach. The patient desired the inlay retainer FRC method. Preparations were made on the abutment teeth using the design parameters described in the previous section (Figures 4–7). The initial slot preparations were modified for an inlay design. Unfortunately, the extent of the initial slot dimensions

Figure 12. *Lingual view of completed FRC bridge.*

Figure 13. *Facial view of completed FRC bridge.*

Figure 14. *Abutment teeth isolated prior to beginning tooth conditioning for adhesive cementation.*

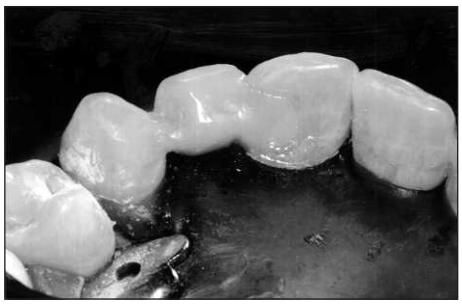


Figure 15. *Lingual view of cemented inlay bridge prior to dam removal.*

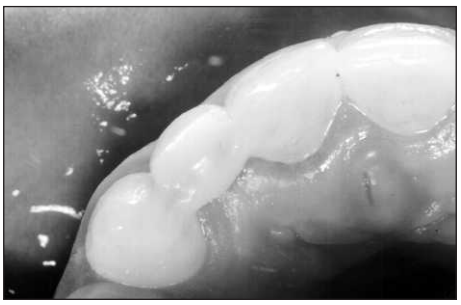


Figure 16. *Lingual view of FRC inlay bridge after rubber dam removal.*

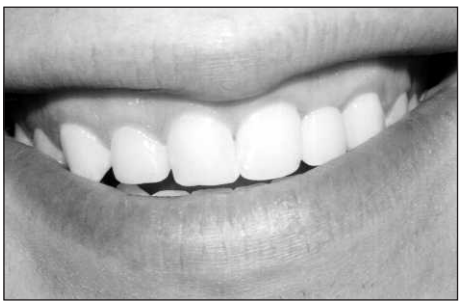


Figure 17. *Facial view of patient after receiving FRC inlay bridge.*

caused the outline form to be a little larger than needed. Additionally, these abutment teeth presented with short clinical crowns, which would have made this case very difficult unless some modifications were made. Electrosurgery was performed around both abutment teeth and on the edentulous ridge to improve the outcome of the case. The left central incisor needed clinical crown lengthening to match the clinical crown height of the right central incisor. Also, the approximal surfaces of the abutment teeth adjacent to the edentulous space needed to be lengthened to permit adequate length of the approximal box preparations and to keep the margins supragingival, and the edentulous ridge was reshaped to allow for a more natural contour of the pontic. An impression of the area was taken with vinylsiloxane impression material in a custom tray. The preparations were temporized using Fermit-N™ (Ivoclar North America), a soft-resin material, and an interim plastic partial was provided to replace the missing central.

PROSTHESIS FABRICATION

This case was fabricated with Sculpture/FibreKor™. Two working casts were fabricated, one with removable dies and a second, solid cast, for use when the FRC substructure was being fabricated (Figure 5). No rubber die spacer was placed, only lubricant. The following describes the laboratory procedures when the Sculpture™/FibreKor™ system was used.

A thin layer of particulate composite was placed on the floor of the preparation of the dies, light polymerized, and then six to seven FRC strips were cut and placed over the particulate layer within each preparation and across the edentulous space (Figure 8). The FRC was polymerized and then an additional 12-15 strips of FRC added to the buccal, lingual, and incisal aspects to create a miniature pontic shape that would provide support for the Sculpture™ particulate composite overlay (Figures 9, 10). The completed anatomic form of the pontic and retainers was developed with particulate composite (Figures 11-13).

CHAIRSIDE DELIVERY

Delivery of FRC bridges involved the following six steps: (1) initial try-in to verify occlusion, anatomic form, approximal contacts, and shade; (2) isolation of the abutment teeth; (3) preparation of the abutment teeth for adhesive cementation–total etch using 34-37% phosphoric acid and application of a fifth-generation one-bottle or fourth-generation multiple-bottle hydrophilic enamel/dentin bonding system; (4) air abrasion with 50µm aluminum oxide at 60–80 psi of the internal aspect of the FRC bridge, providing micromechanical retention for the particulate composite luting cement; (5) cementation using a dual-cured particulate composite resin luting cement; and (6) final adjustments and polishing (Figures 14–17).

CONCLUSION

The development of glass fiber-reinforced composites has made available the fabrication of metal-free, porcelain-free fixed prostheses that can provide esthetic and functional alternatives to all ceramic or porcelain fused-to-metal bridges. This article described the clinical procedures and lab fabrication of an anterior inlay FRC prosthesis. The esthetics that can be achieved with this type of approach are excellent. The ability to adhesively lute these frameworks provides the potential for a conservative intraoral preparation, ideal marginal integrity, and the ability to have nonvisible supragingival marginal designs. Long-term evaluation will determine the eventual place of this technique among the other approaches currently available for replacing missing anterior teeth.

(Received 7 April 1999)

References

Fahl N Jr & Casellini RC (1997) Ceramor/FRC technology: the future of biofunctional adhesive aesthetic dentistry *Signature* **4(2)** 7-13.

Freilich MA, Duncan JP, Meiers JC & Goldberg AJ (1998a) Preimpregnated, fiber-reinforced prostheses. Part I. Basic rationale and complete-coverage and intracoronal fixed partial denture designs *Quintessence International* **29(11)** 689-696.

Freilich MA, Karmaker AC & Burstone CJ (1997) Flexure strength and handling characteristics of fiber-reinforced composites used in prosthodontics *Journal of Dental Research* **76** Abstracts of Papers p 184 Abstract 1361.

Freilich MA, Karmaker AC, Burstone CJ & Goldberg AJ (1998b) Development and clinical applications of a light-polymerized fiber-reinforced composite *Journal of Prosthetic Dentistry* **80(3)** 311-318.

Goldberg AJ & Burstone CJ (1992) The use of continuous fiber reinforcement in dentistry *Dental Materials* **8(3)** 197-202.

Goldberg AJ, Freilich MA, Haser KA & Audi JH (1998) Flexure properties of fiber architecture of commercial fiber reinforced composites *Journal of Dental Research* **77** Abstracts of Papers p 226 Abstract 967.

Zanghellini G (1997) Fiber-reinforced framework and ceromer restorations: a technical review *Signature* **4(1)** 1-5.

Awards

Academy of Operative Dentistry
Award of Excellence
Dr Bruce Brownfield Smith

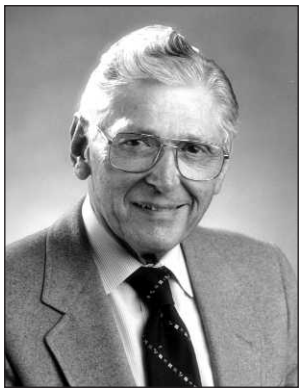
It is a great honor and even greater pleasure that I have been asked to present the Award of Excellence to my friend and colleague, Dr Bruce B Smith.

I first met Bruce when I was a dental student at the University of Washington School of Dentistry. Bruce was one of my teachers. He was a quiet man. We students called him "Whispering Smith" because of his soft voice and quiet, but very firm, manner of teaching. When he stuck the knife to unprepared students, he whispered quietly with his gentle disapproval. He also whispered quietly when he gave approval. He never tolerated students who were unprepared and he never let the excellent students become arrogant. In his soft, quiet manner of teaching, he expected his students to study and give a 100% effort in the clinic. And, he usually got it.

Bruce was born December 16, 1917 in Seattle, WA of a pioneer family. His father and uncle were both dentists. Bruce's father, Nathan Smith, practiced dentistry until he was 90 and lived to be 100. Graduating from high school in 1936, Bruce spent three years at the University of Washington in pre-dental studies. He graduated with honors, Omicron Kappa Upsilon, from North Pacific Dental College (now the University of Oregon) in 1942, receiving the DMD and BSc degrees.

While waiting to be called to active duty with the Naval Reserve, Bruce spent one year teaching at North Pacific Dental College. Following the war, he entered private practice in Seattle and later became one of the original part-time faculty members at the then new University of Washington School of Dentistry, teaching Operative Dentistry, Crown and Bridge, and Ceramics.

Bruce served as mentor of the John Kuratli Study Club (Crown and Bridge) in Oregon, for 18 years and was a member of the University-Ferrier Gold Foil Study Club from 1949 through 1985. He was a founder and first president of the American Academy of Gold Foil Operators (1952 and 1953) and received fellowship in the American College of Dentists and the International College of Dentists in 1953. He is a member of the Pierre Fauchard Society. He has served as vice-chairman and chairman of the ADA section in Operative Dentistry (1976). He was president of the Academy of Operative Dentistry in 1974, as well as president of the American Academy of Restorative Dentistry in 1978-79. He is a member of CAIC dental study seminar and recently received the 1998-99 Seattle King County Dental Society Service Award in honor of his 57 years of dental practice.



Bruce Brownfield Smith

Dr Smith has published several papers in dental literature and produced a movie, Practical Rubber Dam Application, with Dr Gerald D Stibbs, which won an award at the French Film Festival. He has contributed to various operative texts and was the first in the US to organize a closed-circuit TV presentation of chair dental operations for the Washington State meeting in Seattle. He has been an instructor of a number of two-week postgraduate courses in Ferrier gold foil procedures as well as a three-day course in Class II gold foil operations with Dr Stibbs. He has given several courses and lectured on the use of dental porcelain throughout the US and many foreign countries. Bruce has designed several special instruments for use in gold foil operations. He presently is mentor to the George Ellsperman Gold Foil Study Seminar in Seattle.

Bruce was honored with the Distinguished Member award of the American Academy of Gold Foil Operators in 1983. He is licensed to practiced dentistry in Washington, California, and Oregon.

For recreation, Bruce engaged in sailboat racing for 20 years. He served as a summit guide at Mt Rainier ('37-'38) and in 1937 was a member of the Mt Baker avalanche rescue effort featured in *Life Magazine*.

Bruce is a rich man. When a man has two daughters like Joy and Nicki, he is rich. When he has a wife like Lola, he is rich. When a man has parents who loved and cared about him, he is rich. When a man has a distinguished career in a profession that he loves, he is rich. And, when a man can excel with all of his God-given talents, he is rich, indeed.

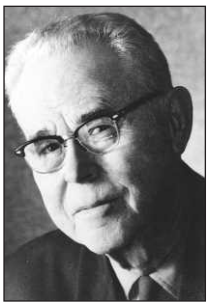
On behalf of the members of the Academy of Operative Dentistry, I present Bruce Brownfield Smith the year 2000 Award of Excellence.

J Martin Anderson

Academy of Operative Dentistry

Hollenback Memorial Prize

Dr Frederick C Eichmiller



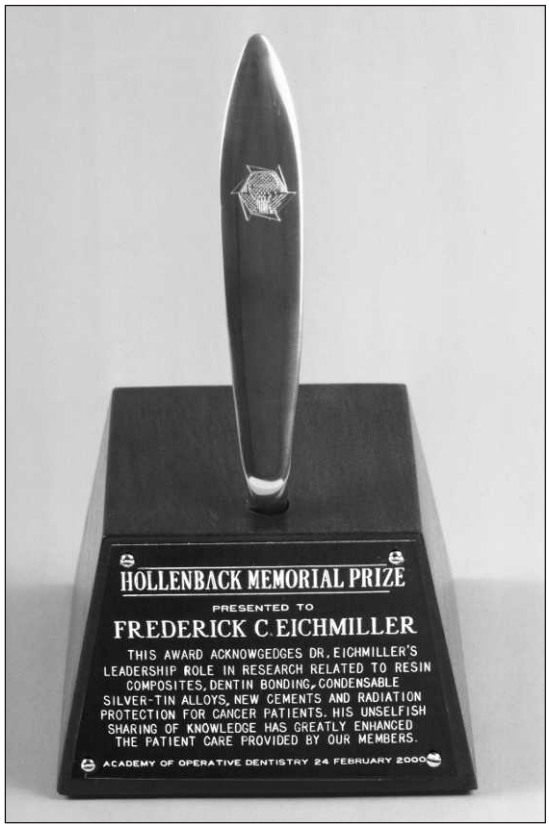
George Hollenback

Frederick C Eichmiller

The Hollenback Memorial Prize is generally given to those dental professionals who are at the end of their careers, but it is to Dr Eichmiller's credit that the Academy of Operative presents this award to him while he is in his prime.

Dr Eichmiller is the Director of the ADA Health Foundation, Paffenbarger Research Center, National Institute of Standards and Technology. This prestigious Institute has undertaken extensive research, which has provided dentistry with many noteworthy concepts and scientific technology. Under his guidance, the Institute continues to be a leader in innovative and creative studies. Dr Eichmiller has a background in engineering as well as dentistry, and this match has served him and the profession well. He has published more than 160 papers and abstracts, holds a number of patents, and continues to work on composite materials, dentin bonding, condensable silver-tin alloys, new cements, and shielding for cancer patients undergoing radiation therapy.

Dr Eichmiller has a gift for bringing this new technology into dental practice. In addition he is active in clinical studies of gold alloys, desensitizing agents, and processes for efficient and effective delivery of dental care. He is an active member of the George M Hollenback and US Navy Dental School Operative Dentistry Study Clubs. His leadership not only of the ADA Institute, but also in his many other activities is much appreciated, and the profession and our patients have greatly benefited from his tireless efforts.



The Academy of Operative Dentistry takes great pleasure in awarding Dr Frederick C Eichmiller the Hollenback Memorial Prize for 2000.

J W Osborne

International Symposium on Management Alternatives for Carious Lesions September 15-17, 2000 Charleston, South Carolina

Registration fee for the three-day conference is:
$295 ($125 for one day)

Phone: (800) 651-2926 or (843) 792-3777
Fax: (843) 792-1107
Web Site: http://www2.musc.edu/cl/home.htm
E-mail: chinnijw@musc.edu
Mail: Office of Continuing Professional Development
MUSC College of Health Professions
PO Box 250822, 19 Hagood Ave, Suite 910
Charleston, SC 29425

The traditional, surgical model for managing dental decay includes excavation of the carious tooth structure followed by restoration with various dental materials. Recently, alternative models have been proposed. Caries is now viewed as an infectious disease process, and a medical model of treatment has been advocated. Non-restorative approaches, such as biomimetics, remineralization, vaccines, and gene replacement therapy are being explored. In the restorative area, minimal intervention has become the watch-word, and non-metallic materials, such as composite resin, glass ionomers, and ceramics are being used in combination with advanced adhesives to provide a conservative, restorative approach.

This program brings together 20 of the world's most renowned clinicians, scientists, and dental educators to review every aspect of the management of dental decay, including prevention, present and future restorative materials, and non-invasive, biologically-based therapy. This program has the potential to direct future academic curricula, research, and delivery of dental care. It is a conference that will be of value to all dental professionals. The papers presented will be published as a special supplement of *Operative Dentistry*.

Symposium Agenda

Friday, September 15, 2000

7:00-10:00	Registration
7:45-8:00	Introduction and Welcome *W Dan Sneed*
Theme 1:	**Non-Restorative Approaches**
8:00-8:50	Current concepts of dental caries and its prevention: The role of third party insurers *Maxwell H Anderson*
8:55-9:45	Risk assessment *Kenneth J Anusavice*
9:45-10:00	Break
10:00-10:50	Remineralization *Lawrence C Chow*
10:55-11:45	Gene replacement therapy *Jeffrey D Hillman*
11:50-12:40	Potential for vaccines in the prevention of carious lesions *Michael W Russell*
12:40-1:00	Panel Discussion – (Research, Treatment Decisions, Caries Risk, Probiotics)
1:00-2:00	Lunch
2:00-2:50	Ways to remove roadblocks to advancement and implementation of knowledge in the field of biomimetics *Norman S Braveman*
2:55-3:45	Assessment of current approaches to dental education *Mark A Latta*
3:50-4:40	Ways underserved populations could benefit from new approaches *Charles R Hook*
4:45-5:00	Panel Discussion – (Barriers to Changing Practice and Education Related to Non-restorative Techniques)

Saturday, September 16, 2000

Theme 2:	**Metallic Restorative Materials and Historical Standards**
8:00-8:50	Performance standards for competitive dental materials *E Steven Duke*
8:55-9:45	Mercury, its impact on the environment and its biocompatibility *John Osborne*
9:45-10:00	Break
10:00-10:50	Gold as a historic standard & role for the future *Cleveland T Smith*
10:55-11:45	Research into non-Hg containing metallic alternatives *Frederick Eichmiller*
11:45-12 noon	Panel Discussion – (Amalgam vs. Non-Hg Containing Alternatives)
12 noon -1:00	Lunch
Theme 3:	**Conservation Dentistry Through Adhesion and Non-Metallic Materials**
1:00-1:50	Adhesives & cements to promote preservation dentistry *Bart Van Meerbeek*
1:55-2:45	Recent commercial composite formulations *M Mike Suzuk*i
2:45-3:00	Break
3:00-3:50	Indirect resin & ceramic systems *Anne Peutzfeldt*
3:55-4:45	Various forms of inonomers *Reinhard A Hickel*
4:45-5:00	Panel Discussion – *Ed Swift* (Are Non-metallic Restoratives Viable as Amalgam Replacements?)

Sunday, September 17, 2000

Theme 4:	**Future Materials and Biocompatibility:**
8:00-8:50	Direct posterior composite restorations *Didier Dietschi*
8:55-9:45	Future polymers *Jack L Ferracane*
9:45-10:00	Break
10:00-10:50	Future ceramic systems *Jean-Francois Roulet*
10:55-11:45	Biocompatibility of restorative materials *Arne Hensten Pettersen*
11:45-12 noon	Panel discussion – *Dorothy McComb* (Future Materials Development to Promote Conservation and Prevention)
12:00-12:30	Directions for future research/Summary *Ivar A Mjör*

The organizers of this symposium would like to acknowledge and express their appreciation for:

- The Sponsorship of Dentsply/Caulk, Espe, Hereaus Kulzer, The Medical University of South Carolina, and The Founders Fund of the Academy of Operative Dentistry.
- The Patronage of GC America, Ivoclar, Shofu, Sybron/Kerr, 3M, and Ultradent.
- Grants from The Centers for Disease Control and The National Institute of Dental And Craniofacial Research.

Departments

Classifieds: Faculty Positions

Operative Dentistry accepts appropriate classified advertisements from institutions and individuals. Advertisements are run at the following rate: $45.00 for 30 or fewer words, plus $0.75 for each additional word. Consecutively repeated ads are run at a flat rate of $50.00. *Operative Dentistry* neither investigates the offers being made, nor assumes any responsibility concerning them, and it reserves the right to edit copy and to accept, delete, or withdraw classified advertisements at its discretion. To ensure publication in a given issue, copy must be received 45 days before the publication date. In other words, copy should be received by 15 November of the preceding year for the January-February issue, and by 15 January, March, May, July, and/or September for publication in subsequent issues. Send advertisements to the editorial office identified inside the front cover.

University of Florida
Operative Dentistry

The University of Florida College of Dentistry invites applications for a full-time, tenure-track faculty position in the Department of Operative Dentistry. Responsibilities include clinical and/or preclinical teaching. Participation in faculty practice is expected, as is research and/or other scholarly activity that results in an enhanced national reputation. A dental degree from an ADA-accredited dental school, or the equivalent, and advanced dental education are preferred. Research experience in adhesive dentistry and extensive clinical experience in direct and indirect tooth-colored restorations is preferred. Salary and academic rank will be commensurate with qualifications. Women and minorities are encouraged to apply. This selection process will be conducted under Florida's "Government in the Sunshine" and Public Record Law. Please submit curriculum vitae to Dr Paul K Blaser, Chair, Search Committee; University of Florida–Health Science Center; PO Box 100415; Gainesville, Florida, 32610-0415 by June 1, 2000. EEO/AA/EA employer.

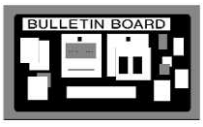
University of the Pacific – Operative Dentistry

The University of the Pacific School of Dentistry in San Francisco is accepting applications for a full-time, tenure-track position at the assistant of associate professor level. Responsibilities will include clinical and preclinical instruction (leading to course directorship). Participation in research is expected and intramural dental practice is available. Applicants should write to: Dr James Simon, Chair, Department of Operative Dentistry, UOP 2155 Webster St, San Francisco, CA 94115 and should enclose a letter of intent showing qualifications, describing career goals, and a curriculum vitae.

University of Iowa – Chair, Department of Operative Dentistry

The University of Iowa's College of Dentistry is presently conducting a search to fill a full-time tenure-track faculty position as Chair of the Department of Operative Dentistry at the rank of Associate or full professor. Major responsibilities include: providing strong departmental leadership, faculty development and mentoring, and maintaining intra-collegiate and university collaborations. The position will be available immediately; screening will begin immediately. Applicants must have a DDS/DMD degree or equivalent, advanced education training in operative dentistry or related field, a record of scholarship; evidence of effective teaching, and relevant administrative experience. Academic rank and salary will be commensurate with qualifications and experience. Submit CV and three letters of recommendation to Search Chair, Dr Georgia Johnson, Department of Periodontics, College of Dentistry, University of Iowa, Iowa City, IA 52242. The University of Iowa is an affirmative action/equal opportunity employer; women and minorities are encouraged to apply.

Announcements



American Academy of Gold Foil Operators
Annual Meeting

1-4 November, 2000
Honolulu, Hawaii

It's not too early to begin planning for your Hawaiian Luau! A one-half day clinical session and two half-day essay sessions are planned. Headquarters Hotel will be the fabulous JW Marriott Ihilani Resort and Spa. For more information, contact: Dr Ronald Harris, AAGFO Secretary-Treasurer, 17922 Tallgrass Court, Noblesville, IN 46060, phone: 317-867-0414, fax: 317-867-3011, or e-mail: piperon@earthlink.com.

Tucker Institute Clinical Course

A clinical course in conservative gold castings, mentored by Dr Richard V Tucker, will be held June 12-16, 2000 at the University of Washington Dental School. For course information, please contact Dr Dennis Miya at 206-244-1618; Fax 206-431-9800.

To Our Contributors and Readers

Operative Dentistry has been extremely fortunate in establishing a reputation for quality that has resulted in increased manuscript submissions each year. We have seen our popularity grow to the point where we received 110 articles for review in 1999. However, while this is an outstanding achievement for our journal, we have developed a backlog of accepted articles that has put us in a position of being 18+ months from acceptance of a manuscript to its publication. As editor, this is an unacceptable length of time in our rapidly changing profession. It is too long for authors to wait for their work to appear in print, and too long for our readers to wait for timely scientific information. Therefore, we have expanded *Operative Dentistry* from 72 to 104 pages for the remainder of 2000. This should allow us to "catch up" and eliminate our backlog of accepted papers. Our subscribers should note that this is being done at no increase in cost to them, thanks to our new Corporate Sponsorship program that provides the necessary funds to cover the additional printing and mailing costs. If our submission rate continues to rise, we will probably continue to publish a larger journal. Our hope is that you will share the news of the expanded issues, encourage your colleagues to subscribe, as well as submit their research to *Operative Dentistry*. Our editorial team's goal is that once a manuscript has been accepted, it will be published within 10 months.

Michael A Cochran, Editor

Operative Dentistry Home Page

We hope all our readers will take advantage of the information available by accessing our Internet home page. Our address is: **http://www.jopdent.org/**

The home page contains a search engine and buttons that, hopefully, will lead you to answers to any questions you may have related to *Operative Dentistry*. These are:

Journal: leads to information on the Editorial Staff and Editorial Board; a complete index of journal volumes; a compilation of direct gold references; highlights or the current, next, and future issues, as well as a more detailed look at published Editorials and Clinical Pearls.

Subscribe: leads to complete information on subscription rates; purchasing back issues, reprints, and bound volumes; and subscription and change of address forms.

Affiliates: provides links to the American Academy of Gold Foil Operators, the Academy of Operative Dentistry, the AADS-Operative Section, and our Corporate Sponsors. In addition, membership applications for the journal's parent academies are available for downloading.

News: announcements of interest to our readers, including meeting information, advertised faculty positions, and upcoming CE courses.

Forum: a message board to allow questions, discussion, and interchange of ideas on operative dentistry.

Authors: complete instructions for contributors to the journal.

Reviewers: password-protected link for our Editorial Board to submit manuscript reviews electronically.

Corporate Sponsorship

Operative Dentistry invites dental manufacturers that share our commitment to timely publication of research and clinical articles relevant to the discipline of restorative dentistry to become Corporate Sponsors of the journal. *Operative Dentistry* is distributed in 54 countries with more than 1,800 subscribers. The cost of sponsorship is U.S. $3,000.00 per year (January through December). Sponsors will be recognized by having their company logo displayed on a special page in each issue of the journal for the duration of their sponsorship. A complimentary subscription to *Operative Dentistry* will be sent to the corporate contact person, and the sponsor's logo will appear in the Corporate Sponsors section of our web page, where it will act as a link to their company's web site. Interested parties should contact the Editor at *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 West Michigan Street, Indianapolis, IN 46202-5186; Phone: (317) 278-4800; Fax: (317) 278-4900; e-mail: editor@jopdent.org; URL: http://www.jopdent.org/

OPERATIVE DENTISTRY

CORPORATE SPONSORS

These Dental Manufacturers have joined *Operative Dentistry* in our commitment to publish quality dental literature in a timely manner. We thank them for their support.

SDI

SOUTHERN DENTAL INDUSTRIES

IVOCLAR

IVOCLAR NORTH AMERICA, INC.

DENTSPLY

CAULK

U2®

ULTRADENT

Den-Mat®

BISCO

Specialists in Adhesive and Composite Technology.

Please view the Corporate Sponsors Page at our website (http://www.jopdent.org) for direct links to these companies.

MAY/JUNE 2000 • VOLUME 25 • NUMBER 3 • 145-248

10-9385 Periodicals
Operative Dentistry
Indiana University School of Dentistry, Rm. S411
1121 West Michigan Street
Indianapolis, IN 46202-5186 USA