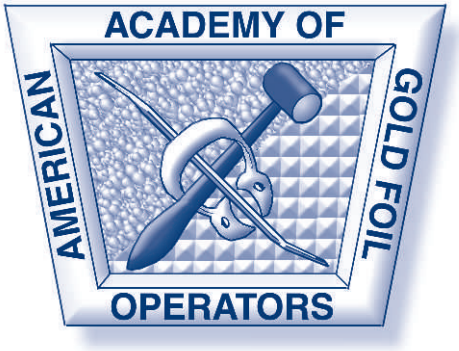
OPERATIVE DENTISTRY



march/april 2004 • volume 29 • number 2 • 121-240

(ISSN 0361-7734)

OPERATIVE DENTISTRY

MARCH/APRIL 2004 • VOLUME 29 • NUMBER 2 • 121-240

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.

Subscriptions: Fax (317) 852-3162

Operative Dentistry (ISSN 0361-7734) is published bimonthly 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.

Current pricing for individual, institutional and dental student subscriptions (both USA and all other countries) can be found at our website: www.jopdent.org, or by contacting our subscription manager:

Fax 317/852-3162
E-mail: jmatis@indy.rr.com

Information on single copies, back issues and reprints is also available. Make remittances payable (in US dollars only) to *Operative Dentistry* and send to the above address. Credit card payment (Visa, MasterCard) 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/

Editorial Staff

Editorial Board

©Operative Dentistry, 2004, **29-2**, 121-122

Editorial

Operative Dentistry: Déjà vu, Redux

The foundation for all that exists in dentistry today was laid with the publication of GV Black's *A Work on Operative Dentistry in Two Volumes* in 1908. This publication (Black, 1908) organized and described the then existing body of knowledge related to the entire field of dental practice. Dr Black's extensive descriptions of the dental supporting tissues, dental pulp, saliva, dental caries, systemic disorders, pathology, oral hygiene, surgery, cavity preparation and dental materials inspired research and scientific investigation and gave birth to today's varied specialties and sub-specialties that were once an integral part of Operative Dentistry. Greene Vardiman Black has been aptly named the "Father of Modern Dentistry."

Unfortunately (depending on your own perspective, of course), the very specialties conceived in the discipline of Operative Dentistry have grown and matured to a point that the profession has forgotten their roots and Operative Dentistry has gradually been relegated to "second-class citizen" status in many schools of dentistry and in the minds of much of the practicing, academic and research communities. The most discouraging aspect is that those of us who claim the discipline seem to somehow believe that we deserve this reduction in status and are going "quietly into that good night" with only a few private grumblings. Programs designed to provide advanced training to the general practitioner (AEGD's and GPR's) spend most of their effort on the specialty areas, following the premise that we learn all we need to know about Operative Dentistry in the pre-doctoral curriculum. This is hard to rationalize since curricular time allotted to Operative Dentistry has been drastically reduced over the years, and much of the clinical "teaching" is done by new graduates with no real clinical experience to offer. Even organizations devoted to Operative Dentistry (AOD, CODE, Operative Section of ADEA, etc) seem loathe to stand up and complain as Departments of Operative Dentistry disappear from our institutions of learning and Divisions of Operative Dentistry in larger Restorative Departments are being downsized and renamed General or Comprehensive Dental Units. This has been a very gradual, erosive process that seems to suddenly have reached crisis proportions despite all the warnings that have been voiced over the years, particularly in this journal by its editors (Hamilton, 1976, 1978, 1983; Bales, 1986, 1988, 1989, 2000; Anderson, 2000; McCoy, 1996, 1997; Cochran, 1995), in Guest Editorials (Wilson & Mjör, 1996; Reinhardt, 1997; Lund, 2001) and in two published supplements (Supplement 1, 1977; Supplement 2, 1981). The current environment in education and organized dentistry is even more surprising when you consider the fact that the procedures integral to Operative Dentistry still comprise the major treatment effort of general practitioners.

On February 12, 1972, Dr David Grainger presented the keynote address at the organizational meeting of the fledgling Academy of Operative Dentistry. His speech was titled "What Are You Operative Dentistry and Why Are They Saying All Those Nasty Things About You?" (Grainger, 1972). Dr Grainger's words were designed to inspire and provide direction for the new Academy of Operative Dentistry, and I would strongly urge all members of the Academy, and anyone with a vested interest in Operative Dentistry, to read this paper (it is posted on the *Journal* website). Dr Grainger's words have been repeated and paraphrased frequently because of their eloquence. In his address, Dr Grainger summarized his feelings in the following manner:

> Dentistry is now in need of men with a different kind of expertise than can be provided by this Academy. Toffler (1970) calls them "multispecialists" as distinct from "monospecialists." These are "men who know one field deeply, but who can cross over into another as well." The creation of the Academy of General Dentistry nurtured such a possibility…but this was asking too much of a group of dedicated men who could and can superbly coordinate a myriad of

monospecialities but could not, by virtue of their roles, know any one to a depth which made them multispecialists; broad ranging men, yes; with depth and crossover, no.

So a need for the last building block still exists—something for the future to fill the void which has been created by a concentration of the specialties at the expense of their original source. Operative Dentistry created dentistry and lost itself in the process. We are faced with an Operative Dentistry that has grown tired, tired from the loss of excitement, from the loss of prestige, from the loss of dignity and even the loss of pride.

The specialties are strong. They have forgotten their humble beginnings and now flex muscles in both the public and private sectors to the benefit of all that is dentistry. To walk with them, Operative Dentistry must look at its fundamentals in the light of the future. Operative Dentistry must accept the challenge of a restorative and biologic multispecialty. The discipline of Operative Dentistry must shrug off its apathy, recognize its worth and dominate once more the direction that dentistry must take. Let us move Greene Vardiman Black into the next lifetime, give him back his energy in an environment that demands a forward look at this, our world of mouths and teeth and tissues. We are skilled in producing skeptics, but not very good at producing individuals who can create their own framework for values. The first step towards the reconstruction of professional values is the rediscovery of values in one's own tradition (Gardner, 1970). GV Black gave us that tradition one lifetime ago.

Dentistry, all dentistry, needs a common purpose, a binding principle some instrumentality for insuring that common goals and excellence are in fact accomplished. What is more logical than Operative Dentistry?…What then are you Operative Dentistry? You are the multispecialty of the future…the hub of the wheel…this Academy.

Despite its age, the message is as valid today as when it was written. It is a message that has been frequently repeated, but seems to fall on deaf ears or is heard by people who are too busy to really listen. As I write this, I can almost hear Yogi Berra saying "It's like déjà vu all over again." If this apathy and inattention continues, the words Operative Dentistry and the discipline they represent will fade into oblivion and appear only as an historical footnote for future generations. Obviously, I am concerned and I hope this editorial raises the same concern in you as you read it. But concern is not enough. Individually and through our alumni groups and academic organizations we must reeducate our schools and the academic community to the importance of Operative Dentistry. As private practitioners, we need to recognize the value of this discipline and garner support from local, state and national dental societies. Our Academy needs to become much more vocal and supportive of our namesake. For once, we need to really hear Dr Grainger's message, recognize our heritage and act positively and enthusiastically to claim our place in the future of our profession. Operative Dentists, stand up with pride and be heard! I sincerely hope that in 2025 I won't be joining all of you at the annual meeting of the Academy of Comprehensive Esthetic Stomatology.

Michael A Cochran
Editor

References

Anderson MH (2000) Progress and excellence *Operative Dentistry* **25(4)** 249-250.

Bales DJ (1986) Restorative Dentistry: Chairmanship *Operative Dentistry* **11(2)** 41.

Bales DJ (1988) Scholarly activities: Boon or bane? *Operative Dentistry* **13(1)** 1.

Bales DJ (1989) Scholarly activities: Part 2 *Operative Dentistry* **14(2)** 57.

Bales DJ (2000) Operative Dentistry in the academic world *Operative Dentistry* **25(3)** 147-148.

Black GV (1908) *A Work on Operative Dentistry in Two Volumes* Medico-Dental Publishing Co Chicago Illinois.

Cochran MA (1995) Operative Dentistry education—The pendulum…and the pit *Operative Dentistry* **20(6)** 217.

Gardner JW (1970) *The Recovery of Confidence* WW Norton New York.

Grainger DA (1972) What are you Operative Dentistry and why are they saying all those nasty things about you? *Journal of the American Academy of Gold Foil Operators* **15(2)** 67-73.

Hamilton AI (1976) Operative instruction—Adequate or not? *Operative Dentistry* **1(4)** 129.

Hamilton AI (1978) Are specialists spoiling dental education? *Operative Dentistry* **3(3)** 81.

Hamilton AI (1983) Educational reform *Operative Dentistry* **8(1)** 1.

Lund MR (2001) Operative Dentistry: The vanishing discipline *Operative Dentistry* **26(6)** 529-530.

McCoy RB (1996) Majestic mediocrity *Operative Dentistry* **21(5)** 181.

McCoy RB (1997) The knee jerk virus *Operative Dentistry* **22(4)** 145.

Operative Dentistry Supplement 1 (1977) How long can Operative Dentistry survive without faculty?

Operative Dentistry Supplement 2 (1981) Part 1: Profile of a clinical teacher; Part 2: The need for graduate education in Operative Dentistry.

Reinhardt JW (1997) Why Operative Dentistry? *Operative Dentistry* **22(5)** 193.

Toffler A (1970) *Future Shock* Random House New York.

Wilson NHF & Mjör IA (1996) What are you Operative Dentistry? *Operative Dentistry* **21(1)** 1-3.

©Operative Dentistry, 2004, **29-2,** 123-130

Clinical Research

Clinical Evaluation of Ceramic Inlays and Onlays Fabricated with Two Systems: Two-Year Clinical Follow Up

MJM Coelho Santos • RFL Mondelli
JRP Lauris • MFL Navarro

Clinical Relevance

Two types of ceramic restorations were used in this study and no significant differences were noticed between them. After two years both ceramic systems demonstrated excellent clinical performance.

SUMMARY

This study evaluated the clinical performance of ceramic inlays and onlays made with two systems: sintered (Duceram, Dentsply-Degussa)—D and pressable (IPS Empress, Ivoclar-Vivadent)—IPS after two years. Eighty-six restorations, 44 IPS and 42 D, were cemented into the mouths of 35 patients. Twenty-seven premolars and 59 molars received Class II preparations totaling 33 onlays and 53 inlays. All restorations were cemented with dual-cured resin cement (Variolink II, Ivoclar-Vivadent) and Syntac Classic adhesive under rubber dam. The evaluations were conducted by two independent investigators at the baseline and after one and two years using the modified USPHS criteria. Additionally, radiographs and slides were made. After two years, 100% of the restorations were assessed and all the restorations were considered clinically excellent or acceptable. Among the analyzed criteria, the following received Bravo ratings: marginal discoloration—IPS (31.82%), D (23.81%); marginal integrity—IPS (18.18%), D (11.9%), color match—IPS (4.55%), D (9.52%) and surface texture—IPS (2.27%); D (14.29%). No "Charlie" or "Delta" scores were attributed to the restorations. The results were subjected to the Fisher and McNemar Statistical Tests. No significant differences were noticed between the two ceramic materials. Among the analyzed criteria, only marginal discoloration presented an increased percentage of "Bravo" scores that increased with time for both ceramic materials. Compared with the baseline data, the

Maria Jacinta M Coelho Santos, DDS, MS, PhD, assistant professor, Federal University of Bahia–School of Dentistry, Department of Operative Dentistry, Bahia, Brazil

Rafael Francisco Lia Mondelli, DDS, MS, PhD, assistant professor, Department of Operative Dentistry, Bauru School of Dentistry, University of São Paulo, Bauru, SP, Brazil

José Roberto Pereira Lauris, MS, PhD, assistant professor, Department of Pedodontics, Orthodontics Community Health, Bauru School of Dentistry, University of São Paulo, Bauru, SP, Brazil

*Maria Fidela Lima Navarro, DDS, PhD, dean of Bauru Dental School, University of São Paulo, Bauru, SP, Brazil

*Reprint request: Al Octávio Pinheiro Brisola 9-75, Vila Universitária, CEP 17012-901, Bauru-SP Brazil; e-mail: mflnavar@usp.br

difference was statistically significant ($p<0.05$). No difference was found between inlay and onlay restorations or between restorations placed in premolars or molars. In conclusion, these two types of ceramic materials demonstrated excellent clinical performance after two years.

INTRODUCTION

Several materials designed to fabricate metal-free restorations are available in the marketplace. Among the esthetic restorative materials available, ceramic materials have emerged due to their outstanding optical properties, biocompatibility and durability (Kelly, Nishimura & Campbell, 1996; Peutzfeldt, 2001). Ceramic materials are considered the best for imitating natural tooth appearance and function based on their capacity to reproduce shades and the translucence of dental structures (Dong & others, 1992; Rosenblum & Schulman, 1997). All-ceramic restorations were first used in the 19th century, but due to the lack of an adhesive cementation technique that allows for good adhesion between the ceramic and tooth structure, a high number of clinical failures were detected (Qualtrough, Wilson & Smith, 1990; Friedl & others, 1996).

In 1965, McLean and Hughes provided the first step in developing a high-strength ceramic with the introduction of alumina-reinforced ceramic crowns. However, only after the 1980s were new all-ceramic systems, such as Cerestore (Biomedical) and Dicor (Dentsply) systems, released into the market (Roulet & Janda, 2001). In the last 20 years, several types of all-ceramic systems with high flexural strength values have been developed. Inlay and onlay restorations may be fabricated from the conventional application of a slurry powder onto a refractory die or it may be cast, pressed, infiltrated or milled (Rosenblum & Schulman, 1997).

These new high-strength ceramic systems, used in conjunction with an adhesive cementation technique, have increased the use of this material in anterior and posterior teeth with a high degree of success. Many studies have referred to the clinical performance of ceramic inlays and onlays made by different systems for the short- and long-term. Krejci, Krejci and Lutz (1992) evaluated the clinical behavior of ceramic inlays, IPS Empress, after 1.5 years and reported a 100% success rate. Fradeani, Aquilano and Bassein (1997) investigated 125 IPS Empress inlays and reported a success rate of 95.63% following four years. Krämer and others (1999) examined 96 heat-pressed ceramic inlays and onlays (IPS Empress) cemented with four different resin luting agents. After four years, seven restorations had failed. Three restorations required endodontic treatment and four exhibited fractures. Fuzzi and Rappelli (1998) evaluated the clinical performance of ceramic inlays and onlays made with two sintered systems cemented with a luting composite. After 11 years, some restorations exhibited endodontic problems, fractures and recurrent caries, with a 95% success rate. Molin and Karlsson (2000) compared the clinical longevity of gold inlays with three ceramic systems (CEREC, IPS-Empress and Mirage). After five years, success rates of 92% and 100% were estimated from ceramic materials and gold restorations, respectively. Krämer and others (1999) considered that some factors; such as cavity design; choice of luting agent and occlusal force exerted more influence on clinical longevity of the restorations than the material choice.

This study evaluated the clinical behavior of all-ceramic inlay and onlay restorations (IPS Empress and Duceram) according to United States Public Health Service (USPHS) criteria over two years. The null hypothesis was that no differences would be found between the two systems, as well as between inlay and onlay restorations and those placed in premolars or molars. Reevaluation of these restorations is planned at two-year follow-up intervals until they have been investigated for no less than six years.

METHODS AND MATERIALS

This study involved 86 Class II inlay and onlay restorations fabricated with two different ceramic systems: 42 sintered ceramics (Duceram–Plus/LFC—D, Dentsply-Degussa Dental, Hanau, Germany) and 44 pressable ceramics (IPS Empress–IPS, Ivoclar-Vivadent, Schaan, Liechtenstein). A total of 33 onlays and 53 inlays were made in 27 premolars and 59 molars by one operator to create a standardized cavity preparation (Table 1). In patients that had more than one restoration placed, the two systems were used in an attempt to achieve the same number of each ceramic system in all patients.

Thirty-five patients including 17 females and 18 males with a median age of 33 years, (ranging from 25 to 44 years) who required inlay and onlay restorations were selected for this study. The involved teeth were in occlusal contact. The volunteers underwent a careful case history review and bitewing and periapical radi-

Table 1: *Number of Ceramic Restorations*

	IPS Empress			Duceram			Total
	PM	M	Total	PM	M	Total	
Inlay	11	14	25	10	18	28	53
Onlay	4	15	19	2	12	14	33
Total	15	29	44	12	30	42	86

PM=premolar; M=molar

ographs were taken. Vitality of the teeth was tested with carbon dioxide snow of -72^{0}C.

The following items were considered as exclusion criteria: high caries risk, periodontal disease, the presence of a removable or fixed orthodontic appliance, signs of bruxism or clenching, the absence of more than one unit in the posterior region, poor oral hygiene and pregnancy. All patients were treated at the Bauru Dental School, University of São Paulo, SP, Brazil. They were informed about the research methodology, risks and benefits, and their right to withdraw participation in this research at any time. A written informed consent was signed. The study was carried out according to research norms and guidelines for human beings deriving from Resolution #196 approved on October, 1996 by the National Health Council and Ethics Research Committee from the Bauru Dental School, University of São Paulo, SP, Brazil.

Tooth Preparation

All cavities were prepared according to the general principles for adhesive inlays and onlays (Nasedkin, 1995; Gürel, 2001). The isthmus width was established between 1.5 to 2.0 mm, the pulpal floor depth was between 1.5 to 2.0 mm, the axial wall depth was 1.5 mm, the internal line angles were rounded and the divergence angle of the cavity was approximately 10 to 15°,with no bevel. The undercuts were covered with resin-modified glass ionomer (Vitremer, 3M Dental Products, St Paul, MN, USA) to achieve the cavity form by removing the build-up material in order to preserve sound tooth structure. The tooth was prepared by means of a tapered, rounded diamond tip in high speed #4137 (ISO #025), #4138 (ISO #018) (KG Sorensen Ind Com Ltda, São Paulo, SP, Brazil) with water spray. The enamel margins were subsequently finished using hand instruments (Zerfing chisel, Duflex, SS White, Rio de Janeiro, RJ, Brazil).

Impression and Provisional Restoration Procedures

Full-arch impressions were made with a polyvinylsiloxane material (Express, 3M Dental) from the prepared arches and with irreversible hydrocolloid (Jeltrate–Dentsply International Inc, PA, USA) from the antagonist arches. Both casts were poured with dental stone type IV (Durone, Dentsply). The bite-registration records were made by a polyvinylsiloxane material (Bite Registration, 3M Dental). Two dental ceramists were selected to produce the inlays and onlays, whose shades were selected from the Vita shade guide (Vita Zahnfabrick, Bad Sackingen, Germany).

Provisional restorations were directly fabricated with the use of self-curing acrylic resin (Duralay–Reliance Dental Mfg Co, Worth, IL, USA) and fixed with eugenol-free cement (Temp Bond NE, Keer, Karlsruhe, Germany).

Luting Procedures

The intraoral fit was evaluated under rubber dam and the internal adjustments performed using diamond burs (KG Sorensen) with low speed. When the fit was not considered satisfactory, the restoration was rejected. Only two restorations were repeated.

Following adjustments, the internal surfaces were sandblasted with 50 µm aluminum oxide particles at a pressure of 87 psi (Opiblast, Buffalo Dental Mfg, Inc, NY, NY, USA). These surfaces were then etched with 10% hydrofluoric acid (Dentsply) for 60 seconds, washed and the silane agent (Monobond S, Ivoclar-Vivadent) applied for 60 seconds and dried. The cavity was cleaned with pumice slurry and etched with 35% phosphoric acid gel for 15 seconds, rinsed with water and gently air dried, taking care to avoid desiccation of the tooth substrate. The dentinal surface was treated with a dentin-bonding agent (Syntac Primer and Adhesive, Ivoclar-Vivadent). Subsequently, the cavity preparation and intaglio surface of the ceramic inlays were covered with a layer of bonding agent (Heliobond, Ivoclar-Vivadent) that was air thinned but not light cured. The dual-cured resin cement Variolink II (Ivoclar-Vivadent) was used for the cementation of all inlays and onlays according to the manufacturer's instructions. The same color luting cement was used for all restorations. Polymerization of the luting agent was performed by light curing the restoration from different positions—occlusal, buccal, lingual and proximal surfaces for 60 seconds in each direction (XL2500, 3M Dental Products, St Paul, MN, USA; 570 mW/cm^2).

Finishing Procedures

Excess luting composite was removed and the occlusal contacts adjusted with diamond finishing burs #1190 FF (ISO #010) and #3203 FF(ISO #012) (KG Sorensen), under water cooling. The surfaces were carefully polished with rubber tips (Cerapol Plus—Edenta AG Dental Rotary Instruments, Hauptstrasse, Switzerland) and the final polishing was conducted using felt discs with diamond polishing gel (KG Sorensen).

Evaluation Procedures

One week following placement, the restorations were assessed using mirrors and probes according to the modified USPHS (Ryge, 1980) criteria (Table 2) by two independent investigators calibrated in the use of the system. The investigators did not participate in the clinical procedures and did not know which system was used on the teeth they were evaluating. These investigators were the same at baseline, one-year and two-year recalls. In addition, bitewing radiographs and intraoral photographs were made and impressions taken (Express, 3M Dental). Replicas were then made

(Araldit, Ciba Geigy GmbH, Wehr, Germany) and gold sputtered (Hummer VII, Anatech LTD, Alexandria, VA), and the occlusal luting interfaces (ceramic/resin cement/enamel) were examined under a scanning electron microscope (JEOL JSM–T220A, Japan) at 100x and 200x magnification. The same procedures performed at baseline were performed at one and two years. Statistical analysis was carried out with Fisher and McNemar tests at a 0.05 level of significance.

RESULTS

All restorations were evaluated at baseline and after one and two years by two independent investigators calibrated in the use of the modified USPHS criteria. The determination of inter-examiner reliability between the two dentists was verified by a Kappa test for each clinical criterion. Surface texture achieved 0.65 agreement at baseline and at one year and 0.82 after two years. Color match presented a Kappa value of 0.78 at baseline, which increased to 0.82 at the following recalls. After two years, marginal integrity and marginal discoloration aspects presented 0.80 and 0.88 of agreement, respectively.

The recall rate was 100% after two years. All the patients informed spontaneously that they were satisfied with the treatment. The Fisher statistical test revealed no significant difference between Duceram and IPS Empress ceramic systems for all aspects evaluated at different recall appointments ($p>0.05$). In Table 3, the results of "Alpha" ratings obtained for both ceramic materials were compared at baseline and at one- and two-year recalls. No recurrent caries or fractures were observed in this period and no Charlie or Delta ratings were detected.

With regard to the clinical behavior of ceramic inlay and onlay restorations and those placed in premolar or molar regions, the Fisher Test showed no statistical difference ($p>0.05$).

The McNemar statistical test was used to draw a comparison between baseline and one year and between

Table 2: *Modified USPHS Criteria for the Clinical Evaluation of Ceramic Inlays and Onlays Used in This Study*

Characteristic	Rating	Criteria
Postoperative sensitivity	**Alpha**	No post-operative sensitivity
	Bravo	Post-operative sensitivity
Secondary caries	**Alpha**	No evidence of caries contiguous with the margin of the restoration
	Bravo	Caries evident contiguous with the margin of the restoration
Marginal discoloration	**Alpha**	No discoloration on the margin between the restoration and the tooth structure
	Bravo	Discoloration on the margin between the restoration and the tooth structure
	Charlie	Discoloration has penetrated along the margin of the restorative material in a pulpal direction
Surface texture	**Alpha**	Smooth surface
	Bravo	Slightly rough or pitted, can be refinished
	Charlie	Rough, cannot be refinished
Marginal integrity	**Alpha**	No visible evidence of ditching along the margin
	Bravo	Visible evidence of ditching along the margin not extending to the DE junction
	Charlie	Dentin or base is exposed along the margin
	Delta	Restoration is mobile, fractured or missing
Color match	**Alpha**	No mismatch in color, shade and translucency between restoration and adjacent tooth structure
	Bravo	Mismatch between restoration and tooth structure within the normal range of color, shade and translucency
	Charlie	Mismatch between restoration and tooth structure outside the normal range of color, shade and translucency
Fracture	**Alpha**	No evidence of fracture
	Bravo	Evidence of fracture

Table 3: *"Alpha" Results of the Clinical Investigation with the Modified USPHS Criteria*

Investigation "Alpha"	Baseline (%)		1 Year (%)		2 Years (%)	
	IPS	D	IPS	D	IPS	D
Post-operative sensitivity	97.73	92.86	100.00	100.00	100.00	100.00
Secondary caries	100.00	100.00	100.00	100.00	100.00	100.00
Fracture	100.00	100.00	100.00	100.00	100.00	100.00
Color match	97.73	90.48	95.45	90.48	95.45	90.48
Marginal discoloration	100.00	100.00	75.00	88.10	68.18	76.19
Marginal integrity	100.00	100.00	88.64	90.48	81.82	88.10
Surface texture	97.73	88.10	97.73	88.10	97.73	85.71

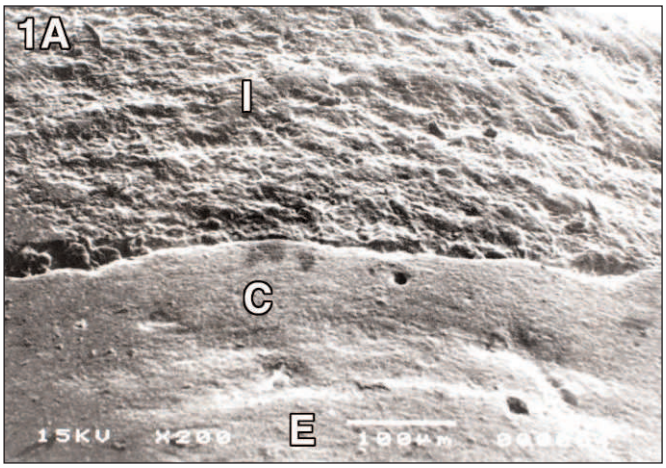


Figure 1A. *Photomicrograph illustrating a perfect margin at baseline; 126I (inlay-IPS Empress), C (resin cement), E (enamel).*

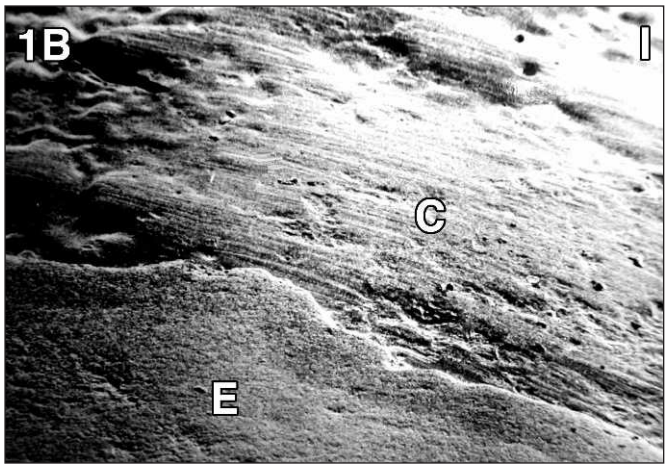


Figure 1B. *Photomicrograph illustrating a perfect margin at baseline; I (inlay-Duceram), C (resin cement), E (enamel).*

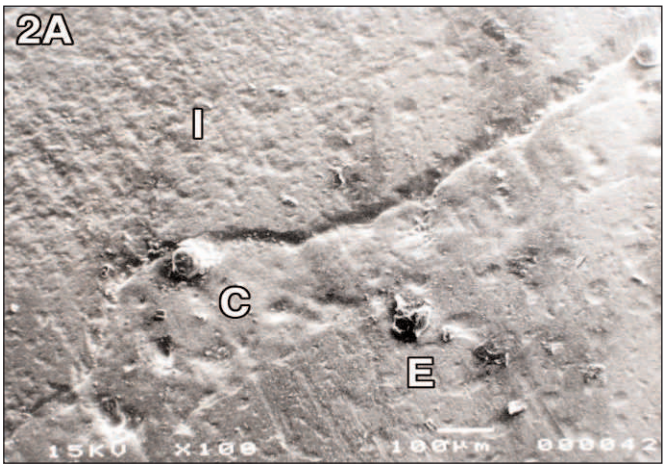


Figure 2A. *Photomicrograph of submargination indicating occlusal wear after one year of function; I (inlay–IPS Empress), C (resin cement), E (enamel).*

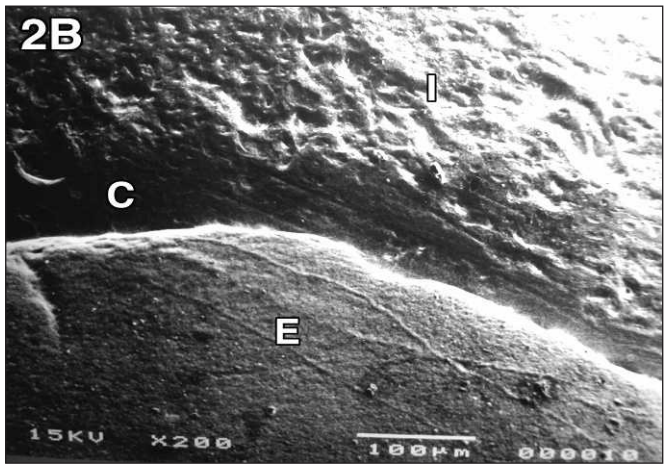


Figure 2B. *Photomicrograph of submargination indicating occlusal wear after one year of function; I (inlay–Duceram), C (resin cement), E (enamel).*

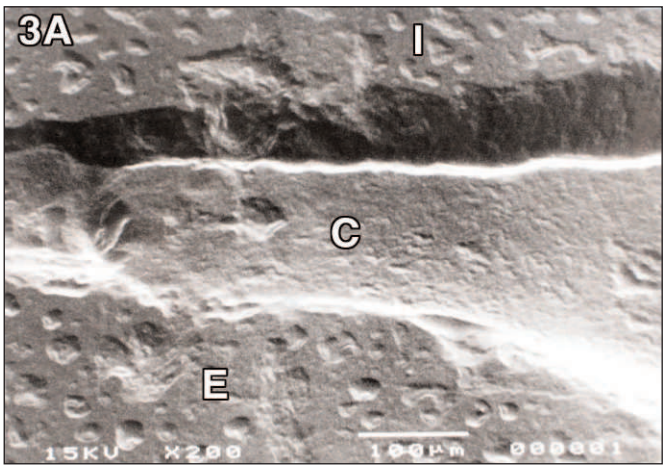


Figure 3A. *Photomicrograph of submargination indicating occlusal wear after two years of function; I (inlay–IPS Empress), C (resin cement), E (enamel).*

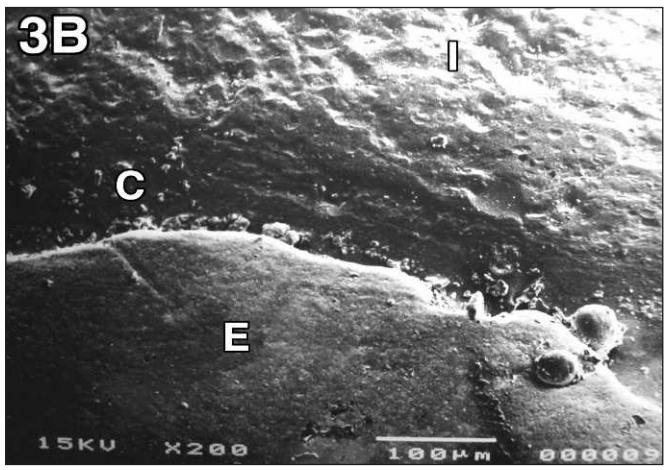


Figure 3B. *Photomicrograph of submargination indicating occlusal wear after two years of function; I (inlay–Duceram), C (resin cement), E (enamel).*

baseline and two years and between one- and two-year recall data for each ceramic system. Significant differences in relation to marginal discoloration were detected between the baseline and one year (p=0.002) and baseline and two-year data (p=0.0005) for IPS Empress ceramic. For the Duceram ceramic, a significant difference was found in relation to marginal discoloration after two years (p=0.004). SEM photographs were taken to show the resin cement wear over time (Figures 1, 2 and 3). The comparison between one- and two-year recall data did not result in significant differences for both ceramic materials. No statistically significant differences were observed after two years regarding postoperative sensitivity, secondary caries, fracture, color match, marginal integrity and surface texture. The estimated survival rate over two years was 100%.

DISCUSSION

The investigated ceramic systems presented satisfactory results after two years of clinical service, with no significant difference between them (p>0.05). The success rate was estimated at 100% for all restorations fabricated with both ceramic systems. These results are in accordance with Friedl and others (1996) and Thonemann and others (1997), whose studies evaluated the clinical performance of ceramic inlays and onlays for the same period. This study presented results superior to those attained by Reinelt and others (1995), whose evaluation of ceramic IPS Empress restorations revealed three inlay failures after two years. One tooth needed to be endodontically treated and two inlays had cohesive fractures. In their study, Tidehag and Gunne (1995) evaluated ceramic IPS Empress inlays and onlays for 26 months and detected fracture in one onlay. Studer and others (1996) reported the results of a two-year investigation of 130 IPS Empress ceramic restorations and presented a success rate of 97.5% with three fractured restorations. It is important to bear in mind that an adequate cavity depth does have a crucial influence on fracture resistance of ceramic inlays and onlays (Thonemann & others, 1997). Another factor predisposing to ceramic fractures may be the type of luting agent used. The strengthening effect of the adhesive cementation technique upon all-ceramic restorations has been reported in several studies (Höglund, Van Dijken & Olofsson, 1992; Anusavice, 1993; Groten & Pröbster, 1997; Sindel & others, 1999; Burke & others, 2002).

In this study, no fractures or recurrent caries were detected after two years. Postoperative sensitivity obtained some "Bravo" ratings at baseline (2.27%-IPS and 7.14%-D), however, it reduced rapidly thereafter. The presence of postoperative sensitivity has been reported in some clinical investigations and is associated with the incomplete sealing of the dentin (Thordrup, Isidor & Hörsted-Bindslev, 1994; Studer & others, 1996; Sorensen & Munksgaard, 1996; Thonemann & others, 1997; Krämer & others, 1999; Frankenberger, Petschelt & Krämer, 2000). In this study, dentin areas near the pulp were protected with calcium hydroxide cement (Dycal, Dentsply) and resin-modified glass ionomer base (Vitremer, 3M Dental) was placed over the pulpal and pulpo-axial cavity floor.

The use of resin-modified glass ionomer (Vitremer) was aimed at blocking out the undercuts, helping to standardize the depth of pulpal floor (from 1.5 to 2.0 mm) and reduce the loss of tooth substance during cavity preparation to achieve divergent walls and preserve dental structure. According to Moscovich and others (1998), a significant amount of dental structure is removed to change a direct restoration into an indirect one.

Marginal discoloration revealed the most significant changes with time for both ceramic materials, showing a statistically significant difference after one year (p=0.002-IPS) and two years (p=0.0005-IPS and p=0.004-D) when the data were compared with baseline. A significant number of clinical investigations (Gladys & others, 1995; Friedl & others, 1996; Thonemann & others, 1997; Fradeani & others, 1997; Krämer & others, 1999; Felden, Schmalz & Hiller, 2000; Frankenberger & others, 2000; Hayashi & others, 2000; Krämer & Frankenberger, 2000; Manhart & others, 2000; Thordrup & others, 2001; Otto & de Nisco, 2002) have shown that marginal discoloration and marginal integrity are the criteria that revealed increased "Bravo" rating, demonstrating that the initial good marginal adaptation does not remain stable in time. However, marginal discoloration does not imply the need for restoration replacement (Krejci & others, 1992; Gladys & others, 1995; Friedl & others, 1996; Thonemann & others, 1997; Krämer & Frankenberger, 2000). These studies showed that wear of the luting composite was the main reason for marginal discoloration and loss of marginal integrity. Van Meerbeek and others (1992) verified, under SEM, a significant decrease in marginal adaptation at the inlay-cement interface after six months of clinical evaluation, indicating severe wear of the resin cement. Zuellig-Singer and Bryant (1998) and Gemalmaz, Özcan and Alkumru (2001) verified higher wear of the luting composite at the cement-ceramic interface compared with the cement-enamel interface. This fact was associated with a very high modulus of elasticity of ceramic which, during chewing forces, transmits stress to the cement whose modulus is lower (Rees & Jacobsen, 1992; Krejci & others, 1992).

The SEM photographs aimed to illustrate the resin cement wear with time. According to Krejci and others, 1992; Gladys and others, 1995; Friedl and others, 1996 and Thonemann and others, 1997, SEM analysis is an efficient instrument to investigate the marginal

integrity and more accurately predict clinical performance of the restorations. However, a clinical examination is still needed to determine whether restoration intervention is necessary.

Concerning the inlay and onlay restoration types and in relation to premolar and molar regions, no statistically significant differences were observed. One hundred percent success ("Alpha" and "Bravo") was achieved for all restorations. Alpha characterized an ideal condition of the restorations, while Bravo was attributed to restorations that presented minor defects with no need for intervention. No "Charlie" or "Delta" ratings were attributed to the restorations. These results are in accordance with Hayashi and others (2000), whose clinical evaluations revealed no significant difference between ceramic inlays placed in premolar and molar regions among all the analyzed criteria. Krämer and Frankenberger (2000) showed no significant difference in resin cement wear between premolar and molar regions. However, Fuzzi and Rappelli (1998), after a 10-year evaluation of 183 sintered ceramic inlays (Microbond and Fortune), showed a success rate of 99% in premolar and 95% in molar regions. Manhart and others (2000) observed significantly higher failure rates for inlays placed in molars compared with premolars. In this study, Bravo ratings for marginal discoloration and marginal integrity were visualized in both restoration types (inlay and onlay) and in both premolar and molar regions, with no statistically significant difference among all aspects assessed. Despite the fact that molars are usually subjected to more intense chewing forces, this negative effect did not show a significant influence at the two-year evaluation recall. So far, inlays and onlays have presented excellent clinical performance in both regions, with no statistical difference between the ceramic systems tested.

CONCLUSIONS

1. Pressed (IPS Empress) and sintered (Duceram) ceramic systems presented similar clinical behavior, showing a 100% survival rate for both ceramic systems in restoring posterior teeth;
2. No recurrent caries or fractures were detected at two years;
3. Marginal discoloration presented statistically significant differences compared with the baseline data; however, there was no need for intervention;
4. No significant differences were found between inlay and onlay restoration types of premolars and molars.

Acknowledgement

The authors thank Ivoclar-Vivadent and Dentsply-Degussa for supporting this study.

(Received 19 March 2003)

References

Anusavice KJ (1993) Recent developments in restorative dental ceramics *Journal of the American Dental Association* **124(2)** 72-84.

Burke FJT, Fleming GJP, Nathanson D & Marquis PM (2002) Are adhesive technologies needed to support ceramics? An assessment of the current evidence *The Journal of Adhesive Dentistry* **4(1-2)** 7-22.

Dong JK, Luthy H, Wohlwend A & Schárer P (1992) Heat-pressed ceramics: Technology and strength *International Journal of Prosthodontics* **5(1)** 9-16.

Felden A, Schmalz G & Hiller KA (2000) Retrospective clinical study and survival analysis on partial ceramic crowns: Results up to 7 years *Clinical Oral Investigations* **4(4)** 199-205.

Fradeani M, Aquilano A & Bassein L (1997) Longitudinal study of pressed glass-ceramic inlays for four and a half years *Journal of Prosthetic Dentistry* **78(4)** 346-353.

Frankenberger R, Petschelt A & Krämer N (2000) Leucite-reinforced glass ceramic inlays and onlays after six years: Clinical behavior *Operative Dentistry* **25(6)** 459-465.

Friedl KH, Schmalz G, Hiller KA & Saller A (1996) *In-vivo* evaluation of a feldspathic ceramic system: 2-year results *Journal of Dentistry* **24(1-2)** 25-31.

Fuzzi M & Rappelli G (1998) Survival rate of ceramic inlays *Journal of Dentistry* **26(7)** 623-626.

Gemalmaz D, Özcan M & Alkumru HN (2001) A clinical evaluation of ceramic inlays bonded with different luting agents *The Journal of Adhesive Dentistry* **3(3)** 273-283.

Gladys S, van Meerbeeck B, Inokoshi S, Willems G, Braem M, Lambrechts P & Vanherle G (1995) Clinical and semi quantitative marginal analysis of four tooth-coloured Inlay systems at 3 years *Journal of Dentistry* **23(6)** 329-338.

Groten M & Pröbster L (1997) The influence of different cementation modes on the fracture resistance of feldspathic ceramic crowns *International Journal of Prosthodontics* **10(2)** 169-177.

Gürel G (2001) Porcelain inlays and onlays *Dental Clinics of North America* **45(1)** 117-125.

Hayashi M, Tsuchitani Y, Kawamura Y, Miura M, Takeshige F & Ebisu S (2000) Eight-year clinical evaluation of fired ceramic inlays *Operative Dentistry* **25(6)** 473-481.

Höglund, C, Van Dijken J & Olofsson AL (1992) A clinical evaluation of adhesively luted ceramic inlays. A two year follow-up study *Swedish Dental Journal* **16(4)** 169-171.

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

Krämer N, Frankenberger R, Pelka M & Petschelt A (1999) IPS Empress inlays and onlays after four years—a clinical study *Journal of Dentistry* **27(5)** 325-331.

Krämer N & Frankenberger R (2000) Leucite-reinforced glass ceramic inlays after six years: Wear of luting composites *Operative Dentistry* **25(6)** 446-472.

Krejci I, Krejci D & Lutz F (1992) Clinical evaluation of a new pressed glass ceramic inlay material over 1.5 years *Quintessence International* **23(3)** 181-186.

Manhart J, Scheibenbogen-Fuchsbrunner A, Chen HY & Hickel R (2000) A 2-year clinical study of composite and ceramic inlays *Clinical Oral Investigations* **4(4)** 192-198.

McLean JW & Hughes TH (1965) The reinforcement of dental porcelain with ceramic oxides *British Dental Journal* **119(6)** 251-267.

Molin MK & Karlsson SL (2000) A randomized 5-year clinical evaluation of 3 ceramic inlay systems *International Journal of Prosthodontics* **13(3)** 194-200.

Moscovich H, Creugers NH, De Kanter RJ & Roeters FJ (1998) Loss of sound tooth structure when replacing amalgam restorations by adhesive inlays *Operative Dentistry* **23(6)** 327-331.

Nasedkin JN (1995) Ceramic inlays and onlays: Update 1995 *Journal of the Canadian Dental Association* **61(8)** 676-681.

Otto T & De Nisco S (2002) Computer-aided direct ceramic restorations: A 10-year prospective clinical study of Cerec CAD/CAM inlays and onlays *International Journal of Prosthodontics* **15(2)** 122-128.

Peutzfeldt A (2001) Indirect resin and ceramic systems *Operative Dentistry* **26(6)** 153-176.

Qualtrough AJ, Wilson NH & Smith GA (1990) Porcelain inlay: A historical view *Operative Dentistry* **15(2)** 61-70.

Rees JS & Jacobsen PH (1992) Stresses generated by luting resins during cementation of composite and ceramic inlays *Journal of Oral Rehabilitation* **19(2)** 115-122.

Reinelt C, Krämer N, Pelka M & Petschelt A (1995) *In vivo* performance of IPS Empress® Inlays and Onlays after two years *Journal of Dental Research* **74** Abstract #1211 p 552.

Rosenblum MA & Schulman A (1997) A review of all-ceramic restorations *Journal of the American Dental Association* **128(3)** 297-307.

Roulet JF & Janda R (2001) Future ceramic systems *Operative Dentistry* **26(6)** 211-228.

Ryge G (1980) Clinical criteria *International Dental Journal* **30(4)** 347-358.

Sindel J, Frankenberger R, Krämer N & Petschelt A (1999) Crack formation of all-ceramic crowns dependent on different core build-up and luting materials *Journal of Dentistry* **27(3)** 175-181.

Sorensen JA & Munksgaard EC (1996) Relative gap formation of resin-cemented ceramic inlays and dentin bonding agents *Journal of Prosthetic Dentistry* **76(4)** 374-378.

Studer S, Lehner C, Brodbeck U & Schärer P (1996) Short-term results of IPS-Empress inlays and onlays *Journal of Prosthodontics* **5(4)** 277-287.

Thonemann B, Federlin M, Schmalz G & Schams A (1997) Clinical evaluation of heat-pressed glass-ceramic inlays *in vivo*: 2 year results *Clinical Oral Investigations* **1(1)** 27-34.

Thordrup M, Isidor F & Hörsted-Bindslev P (1994) A one-year clinical study of indirect and direct composite and ceramic inlays *Scandinavian Journal of Dental Research* **102(3)** 186-192.

Tidehag P & Gunne J (1995) A 2-year clinical follow-up study of IPS Empress ceramic inlays *International Journal of Prosthodontics* **8(5)** 456-460.

Van Meerbeek B, Inokoshi S, Willems G, Noack MJ, Braem M, Lambrechts P, Roulet JF & Vanherle G (1992) Marginal adaptation of four tooth-coloured inlay systems *in vivo Journal of Dentistry* **20(1)** 18-26.

Zuellig-Singer R & Bryant RW (1998) Three-year evaluation of computer-machined ceramic inlays: Influence of luting agent *Quintessence International* **29(9)** 573-582.

©Operative Dentistry, 2004, **29-2**, 131-141

Finishing and Polishing of Indirect Composite and Ceramic Inlays *In-vivo*: Occlusal Surfaces

M Jung • O Wehlen • J Klimek

Clinical Relevance

With respect to margin and surface quality, there were no significant differences between the methods used for finishing and polishing the occlusal surfaces of composite and ceramic inlays *in vivo*. Initial finishing with a 30 µm diamond followed by a tungsten carbide bur caused a significantly greater amount of continuous margins compared to finishing with two diamonds.

SUMMARY

This study evaluated occlusal margins and surfaces of composite and ceramic inlays after finishing and polishing *in vivo*.

Eighty Class II cavities surrounded by enamel were prepared by two experienced dentists. Forty cavities were restored with indirect microhybrid composite inlays (Tetric), the balance were treated with heat-pressed glass ceramic inlays (IPS Empress). Using a rubber dam, the inlays were inserted adhesively with a dual curing composite of high viscosity (Variolink Ultra). Finishing was performed with the sequence of a 30 µm and 20 µm diamond (finishing method FM 1) or a 30 µm diamond followed by a tungsten carbide finishing bur (FM 2). The composite inlays were divided into four groups of 10 that were finished and polished according to the following protocol: (A) FM 2/Diafix-oral, (B) FM 2/MPS gel, (C) FM 1/Diafix-oral, (D) FM 1/MPS gel. Ten ceramic inlays each were treated as follows: (E) FM 2/MPS gel, (F) FM 1/MPS gel, (G) FM 2/ Diamond polisher, (H) FM 1/Ceramiste silicon polishers. After polishing, replicas of the restorations were fabricated. The replicas were examined by SEM with respect to margin quality (portion of continuous margins, overhangs, submargination and marginal imperfections). Furthermore, surface properties were evaluated qualitatively, which included assessing roundness of the contours in three grades (smooth rounding, few edged contours or predominantly edged contours) and evaluation of the surface roughness (smooth and homogeneous surface, minor roughness or severe roughness).

Quantitative analysis of the occlusal composite and ceramic inlay margins showed that 52.2% - 84.6% were rated as continuous, 0% - 14.0% were characterized by overhangs and 0.7% - 10.8% by submargination. A portion of 4.9% - 18.1% margins revealed imperfections. The amount of marginal gap formation was negligible. Composite and

*Martin Jung, priv-doz dr, Dental Clinic, Policlinic for Operative and Preventive Dentistry, Justus-Liebig-University, Giessen, Germany

Oliver Wehlen, DDS, Policlinic for Operative and Preventive Dentistry, Justus-Liebig-University, Giessen, Germany

Joachim Klimek, DDS, professor, Policlinic for Operative and Preventive Dentistry, Justus-Liebig-University, Giessen, Germany

*Reprint request: Schlangenzahl 14, D-35392 Giessen, Germany; e-mail: martin.jung@dentist.med.uni-giessen.de

ceramic inlays showed a similar behavior with respect to marginal quality after finishing and polishing. Overall, there were no significant differences among the four methods applied to composite and the four methods used on ceramic inlays with respect to margin quality. The use of a 30 µm diamond followed by a tungsten carbide bur on composite and ceramic inlays resulted in a significantly larger portion of continuous margins compared to finishing with two diamonds (p=0.049).

Qualitative evaluation of composite and ceramic inlays revealed that 50% - 80% of the occlusal surfaces were characterized by few edged contours and 10% - 50% by smooth rounding. With respect to roughness, smooth surfaces prevailed both on composite (67.5% - 80.0%) and ceramic inlays (64.5% - 77.3%). Overall, no significant differences were detectable between the methods for finishing and polishing composite inlays and the methods applied to ceramic restorations with respect to roundness of contours and surface roughness.

INTRODUCTION

Restoration of posterior teeth with tooth-colored inlays has become a clinically reliable method in aesthetic dentistry. The results of clinical trials with adhesively luted composite or ceramic inlays are promising (Hayashi & others, 2000; Molin & Karlsson, 2000; Pallesen & Van Dijken, 2000).

Several factors that contribute to the success of adhesive inlays have been the subject of many investigations. The effects of different inlay materials (Leirskar & others, 1999; Monaco & others, 2001; Thordrup, Isidor & Horsted-Bindslev, 1999; Van Meerbeek & others, 1992), the role of various luting composites (Gemalmaz, Özcan & Alkumru, 2001; Sjögren, Molin & Van Dijken, 1998; Van Dijken, Hoglund-Aberg & Olofsson, 1998) and the type of dentin conditioning (Thonemann & others, 1994) as well as the method of insertion (Noack, Locke & Roulet, 1993) have been examined. Finishing and polishing have been identified as additional factors that determine the quality and behavior of adhesive restorations (Roulet, 1987).

After adhesive insertion of composite and ceramic inlays, finishing and polishing are necessary for two reasons. Excessive luting composite has to be removed by rotary instruments and, in some instances, occlusal adjusting requires trimming of the restoration surfaces.

Studies that deal with the effects of finishing and polishing on surface quality are mostly performed under experimental conditions using specimens with more or less even surfaces. These studies are necessary and valuable for evaluating the fundamental smoothing efficacy or possible detrimental effects of finishing and polishing techniques on the surface of restorative materials. In contrast, the clinical situation is characterized by several special conditions that cannot be simulated adequately *in vitro*. One aspect is the anatomical situation in the oral cavity. The presence of cusps and fissures on the occlusal surface and the aggravated access to posterior teeth impairs the execution of trimming procedures. Furthermore, the effect of finishing and polishing on restoration and cavity margins and on the luting gap cannot be evaluated under experimental conditions. Currently, publications that examine finishing and polishing techniques under clinical conditions are very rare.

In a previous study, a hybrid composite generally used for the fabrication of inlays and a heat-pressed glass ceramic were subjected to various finishing and polishing procedures under experimental conditions (Jung, 2002). Therefore, this study evaluated the effect of a selection of finishing and polishing techniques on the margins and surfaces of indirect composite and ceramic inlays *in vivo*.

METHODS AND MATERIALS

Eighty Class II cavities were restored with indirect laboratory-manufactured composite and ceramic inlays by two experienced dentists. Luting of all restorations was accomplished with Variolink Ultra (Vivadent, Schaan, Liechtenstein). The composition of this luting composite is shown in Table 1. To finish the occlusal surfaces, diamonds and tungsten carbide burs were used. The first finishing method (FM 1) included a sequence of two finishing diamonds with 30 µm and 20 µm particle size (instruments No 1-6, Table 2). The second finishing method (FM 2) started with 30 µm diamonds (instruments No 1-3) followed by tungsten carbide finishing burs (instruments No 7-9, Table 2). For clinical application, the finishing instruments were mounted in two new red-ring handpieces (24 LN Intra Matic Lux 2, KaVo, D-88400 Biberach, Germany) and were used at 40,000 RPM under three-way water cooling. A new bur was used after five restorations.

Polishing of the occlusal inlay surfaces was accomplished through four different methods:

- Two Striper MPS (Premier Dental Products, Norristown, PA, USA): a diamond impregnated polishing gel
- Diafix-oral (Mueller Dental, D-51789 Lindlar, Germany): a diamond impregnated felt wheel
- Diamond polisher (Brasseler, Savannah, GA, USA): a diamond impregnated rubber polisher in a pointed shape

- Ceramiste (Shofu Inc, Kyoto 605-0983, Japan): a silicon-carbide polishing system used in a pointed and cup shape

These polishing methods were applied according to manufacturer's instructions; details are specified by Jung (2002). Two new blue-ring handpieces 20 LN/68 LDN (KaVo) were used for polishing. Each instrument was only used for one restoration.

The composite inlays were divided into four groups of 10 restorations, which were finished and polished according to four different protocols. Table 3 shows the corresponding combinations of finishing and polishing methods used. Likewise, four groups of 10 ceramic inlays each were formed. The finishing and polishing regimens for the ceramic inlays are also presented in Table 3.

Both the number of composite and ceramic inlays and the methods for finishing and polishing were equally distributed between the two participating dentists. Selection of the finishing and polishing methods followed a randomized protocol.

Clinical Procedure

The realization of the clinical study was approved by the ethic commission of the faculty of medicine of the Justus-Liebig-University Giessen (chairman: Prof Dr E Habermann; Sign: ProfHa/L-10/96).

Patients participating in the study ranged in age from 18-65 years and all teeth were sensitive to thermal stimulation. The cavities were shaped according to the principles of adhesive inlay preparation without any margin beveling. The complete cavity margins were located in enamel. After an impression was taken (Permadyne F Penta and Permadyne Garant Penta 2:1,

Table 1: *Composition of the Luting Composite Variolink Ultra (based on information by manufacturer)*

Component	Base (% by weight)	Catalyst (% by weight)
Bisphenol-A-Glycidyl-dimethacrylate (Bis-GMA)	10.3	10.3
Urethandimethacrylate (UDMA)	5.1	5.1
Triethylenglycoldimethacrylate (TEGDMA)	5.1	5.1
Barium glass (silane treated)	44.2	43.8
Sphaerosil (silane treated)	10.0	10.0
Ytterbiumtrifluoride (YbF3)	25.0	25.0
Pigments	0.04	0.04
Catalysts, stabilizers	0.26	0.66

Table 2: *Details of the Rotary Instruments Used for Finishing (all instruments by Brasseler, Savannah, GA, USA)*

Instrument -#	Type of Bur	Grain Size/ Number of Blades	Shape	Order-#
1	Diamond	24-40 µm	Spherical	806 314 001 514 023
2	Diamond	24-40 µm	Chamfer	806 314 537 514 012
3	Diamond	24-40 µm	Flame	806 314 540 514 009
4	Diamond	15-30 µm	Spherical	806 314 001 504 023
5	Diamond	15-30 µm	Chamfer	806 314 875 EF 012
6	Diamond	15-30 µm	Flame	806 314 889 EF 009
7	Tungsten carbide bur	20	Spherical	500 314 001 071 023
8	Tungsten carbide bur	8	Chamfer	500 314 537 072 012
9	Tungsten carbide bur	12	Flame	500 314 496 071 009

Table 3: *Specification of the Methods Used for Finishing and Polishing Composite and Ceramic Inlays*

Type of Inlay	Finishing Method	Polishing Method
Composite (Tetric; n=10)	30 µm diamond/tungsten carbide bur (FM 2) →	Diafix-oral
Composite (Tetric; n=10)	30 µm diamond/tungsten carbide bur (FM 2) →	MPS gel
Composite (Tetric; n=10)	30 µm/20 µm diamond (FM 1) →	Diafix-oral
Composite (Tetric; n=10)	30 µm/20 µm diamond (FM 1) →	MPS gel
Ceramic (Empress; n=10)	30 µm diamond/tungsten carbide bur (FM 2) →	MPS gel
Ceramic (Empress; n=10)	30 µm diamond/tungsten carbide bur (FM 2) →	Diamond polisher
Ceramic (Empress; n=10)	30 µm/20 µm diamond (FM 1) →	MPS gel
Ceramic (Empress; n=10)	30 µm/20 µm diamond (FM 1) →	Ceramiste

3M ESPE Dental Products, St Paul, MN, USA), the inlays were manufactured in a dental laboratory by one experienced technician.

Table 4: *Significance (p) of Margin Quality and Surface Roughness of Composite and Ceramic Inlays after Rotary Instrumentation (One-way Anova with Respect to "CM"; Kruskal-Wallis Test for the Remaining Criteria); CM – Continuous Margins, OH - Overhangs, SM - Submargination, MI - Marginal Imperfection, SS - Smooth Surfaces, MR - Minor Roughness, SR - Severe Roughness*

	Margin Quality				Surface Roughness		
	CM	OH	SM	MI	SS	MR	SR
Rotary instrumentation of composite inlays (4 methods, n=10 each)	0.325	0.994	0.606	0.811	0.502	0.716	0.498
Rotary instrumentation of ceramic inlays (4 methods, n=10 each)	0.253	0.361	0.606	0.195	0.647	0.783	0.425
Type of inlay Tetric/Empress (n=40 each)	0.565	0.973	0.077	0.117	0.589	0.454	0.470

Forty inlays were made of the micro-hybrid composite Tetric (Vivadent, FL-9494 Schaan, Liechtenstein); another 40 inlays were fabricated using the heat-pressed glass ceramic IPS Empress (Ivoclar, D-73471 Ellwangen, Germany). Details of the material composition are given by Jung (2002).

Adhesive cementation of the inlays was done with the help of rubber-dam isolation. Syntac Primer and Adhesive (Vivadent) were applied for dentin conditioning. Dual-curing Variolink Ultra (Vivadent) was used as the luting composite. Placement of the inlays followed the principles of the ultrasonic insertion technique (Noack & others, 1993). Polymerization was performed with the Optilux 400 curing unit (VCL 401, Demetron/Kerr, Danbury, CT, USA).

The removal of composite overhangs, occlusal adjustment and finishing were performed immediately. The final polishing was accomplished at a separate appointment within 14 days. Upon completion of this appointment, an impression for the fabrication of replicas was taken (Silaplast and Silasoft, Detax, D-76275 Ettlingen, Germany).

Replica impressions were filled with the epoxy-resin Stycast 1266 (Grace NV Specialty Polymers, B-2260 Westerlo, Belgium). After 24 hours of curing, the replicas were prepared for scanning electron microscopy (SEM). After mounting on a special SEM-tray with the adhesive Leit-C (Neubauer chemical drugs, D-48031 Muenster, Germany), the replicas were gold-coated (sputtering device SCD 040, Bal-Tec, FL-9464 Balzers, Liechtenstein). In SEM, the inlay-replicas were examined with respect to quality of margins and surface (Table 4).

Margin Quality

The inlay margins were evaluated using the SEM Amray 1610 (Amray, Bedford, MA, USA). The working tension was set at 10 kV with an emission of 50 µA. The margins of all inlays were evaluated stepwise at an original magnification of 100x. The type of margin quality was graded according to the following five criteria:

- continuous/perfect margin (CM)
- overhangs (OH)
- submargination (SM)
- marginal imperfection (MI)
- marginal gap formation (MG)

For each restoration, the individual length of each of the five different margin criteria was calculated and related to the total margin length (quantitative margin analysis: Win-Mes Version 2.0; Stefan Kueppers, Medical Software Solutions, D-91054 Erlangen, Germany).

Surface Quality

All the restorations placed were examined using a PSEM 500 (Philips Electronics, 5600 MD Eindhoven, Netherlands) at a working tension of 25 kV. The surfaces of each restoration were evaluated qualitatively with the help of three photomicrographs: an overview at an original magnification 10x and two detailed views of cusp and fissure surfaces at a magnification 80x. Photo prints 16x12 cm in size were used for evaluation.

The prints of the overviews were assessed in three categories with respect to roundness of contours:

- smooth rounding
- few edged contours
- predominantly edged contours

The photo prints of the details of cusps and fissures were subdivided into 48 squares; each square was assessed separately for roughness using the following categories:

- smooth surfaces
- minor roughness
- severe roughness

During evaluation of the margins and surfaces, the finishing and polishing methods and inlay materials were blind to the examiner.

Statistical analysis was carried out using SPSS for Windows (Version 7.5.2G, SPSS Inc, Chicago, IL, USA). The data of margin analysis with respect to "continuous margins" were distributed normally and analyzed by one-way ANOVA; the data of the remaining four criteria were subjected to the Kruskal-Wallis test for independent samples. The results of surface evaluation were analyzed with the help of chi²-tests for crosstables (roundness of contours) or the Kruskal-Wallis test for independent samples (roughness).

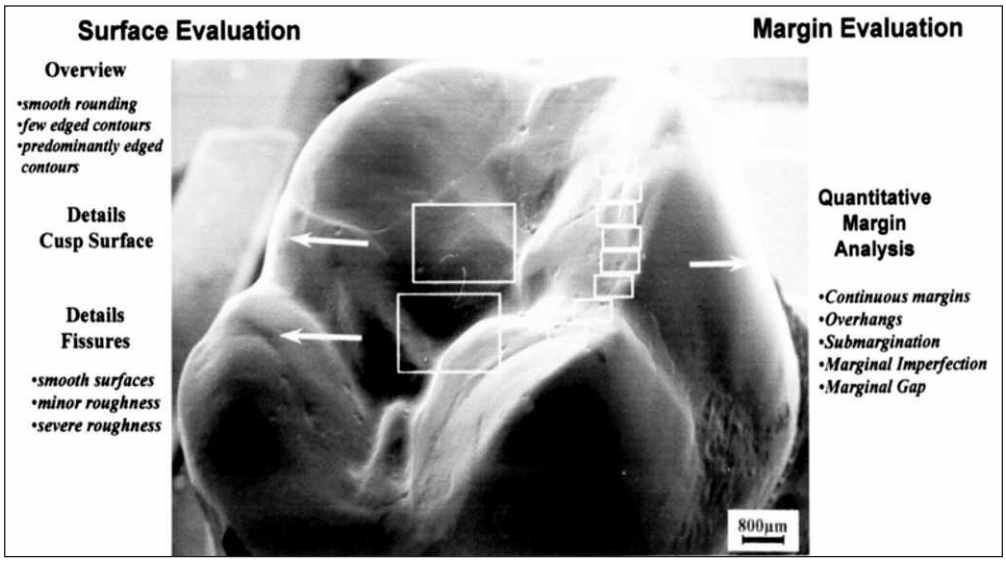


Figure 1. *The evaluation of replicas made from composite and ceramic inlays by SEM with the help of margin analysis and qualitative surface assessment with respect to roundness of contours and roughness.*

RESULTS

Twenty-two patients participated in this clinical study (15 females, 7 males). The average age was 34.3 years. Forty-three premolars and 37 molars were included (51 mo/od restorations, 27 restorations with three surfaces, 2 with four surfaces). Figure 1 shows an example of the replicas used for evaluation. Figure 2 shows distribution of the number of restorations among the posterior teeth.

Margin Quality

Figure 3 illustrates representative examples for the evaluation of composite and ceramic inlays according to four categories of margin quality.

With respect to occlusal surfaces of composite inlays, continuous margins were most frequent (52.2% - 70.0%); 4.5% - 8.2% were rated as overhangs, 0.7% - 7.0% as submargination and 13.0% - 18.1% were rated as imperfect (Figure 4A). There were no marginal gaps. There were no significant differences among the four methods used for finishing and polishing composite inlays with respect to distribution of marginal criteria (Table 4).

The corresponding results for the ceramic inlays showed the same tendency (Figure 4B): 0% - 14.0% of the margins were rated as overhangs, 4.7% - 10.8% by sub-margination, 4.9% - 16.4% by imperfections and 61.0% - 84.6% as perfect margins. There was no marginal gap formation. Again, the four methods applied to the ceramic inlays showed no significant differences for the portion of any of the marginal criteria.

Overall, there were no significant differences between composite and ceramic inlays with respect to distribution of any of the five marginal criteria.

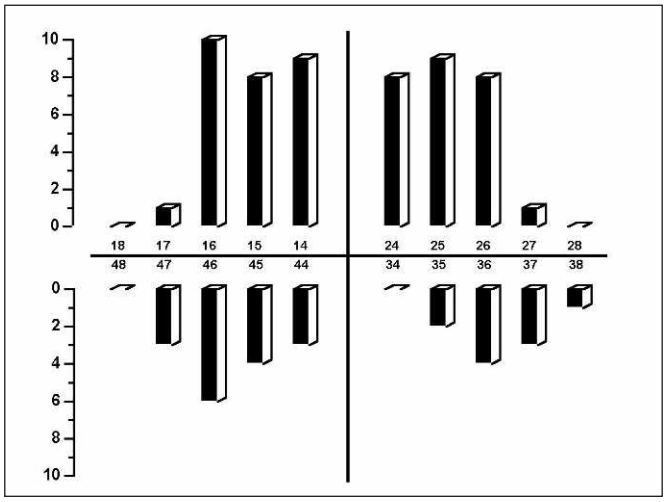


Figure 2. *Number and distribution of 40 composite and 40 ceramic inlays among maxillary and mandibular posterior teeth.*

The method used for finishing had a significant effect on the amount of continuous margins. When occlusal finishing was performed with a diamond followed by a tungsten carbide bur (FM 2), the portion of perfect margins was significantly greater than after finishing with two diamonds (FM 1; p=0.049). Furthermore, there was a tendency toward fewer marginal imperfections when FM 2 was applied.

Surface Quality

Roundness of Occlusal Contours

Smooth rounding was attributed to 20% - 50% of the composite inlays (Figure 5A); most surfaces of the composite inlays were characterized by few edged contours (50% - 80%) and predominantly edged contours were

found only after applying Diafix-oral felt wheels after finishing according to FM 2 (20%). No significant differences were detectable between the methods for finishing and polishing composite inlays with respect to roundness of contours (p=0.29).

The results for the ceramic inlays were similar (Figure 5B). Ten to 50% of the inlays were rounded smoothly; 50% to 80% of the inlays had few edged contours and two of the finishing and polishing methods caused predominantly edged contours on 10% of the ceramic inlays. Again, there were no significant differences among the four methods under evaluation (p=0.53).

Surface Roughness

Qualitative evaluation of surface roughness revealed that smooth surfaces prevailed both on composite (67.5% - 80.0%; Figure 6A) and ceramic inlays (64.5% - 77.3%; Figure 6B). Minor roughness was detected on 15.8% - 20.5% of the composite and 20.0% - 30.3% of the ceramic surfaces. Of the composite inlay surfaces, 4.3 - 12.8% and 2.0% - 8.3% of the ceramic restorations were characterized by severe roughness.

With respect to roughness, there were no significant differences between the methods for finishing and polishing composite and ceramic inlay surfaces. Furthermore, no significant differences could be detected between surface roughness of composite and ceramic inlays.

DISCUSSION

The selection of rotary instruments for finishing and polishing was based on the results of a prior experimental study using specimens of the same two materials as in this clinical investigation (Jung, 2002). According to this investigation, applying MPS polishing gel turned out to be efficient both on composite and ceramic surfaces, whereas the diamond impregnated felt wheel achieved good polishing results only on composite surfaces. The diamond polisher and Ceramiste system were designed for application on ceramic surfaces.

Using either diamonds or tungsten carbide burs for finishing might influence the quality of restoration surfaces and margins (Herrgott, Ziemiecki & Dennison, 1989; Jung, Baumstieger & Klimek, 1997; Kaplan & others, 1996; Ward, Tate & Powers, 1995). Therefore, the methods used to finish the composite and ceramic inlays were selected so that half of the methods included two diamonds and the other half included a sequence of a diamond and a tungsten carbide bur.

Employing replicas for the evaluation of restored teeth is a commonly used technique. An epoxy resin was chosen for fabrication of the replicas, because of its fine grain size, which achieved a high precision and homogeneous quality of the replica surfaces.

Quantitative margin analysis in SEM is an established method for assessing the quality of restorations (Roulet, 1987; Roulet & others, 1989). In many cases, baseline results are compared with the situation after

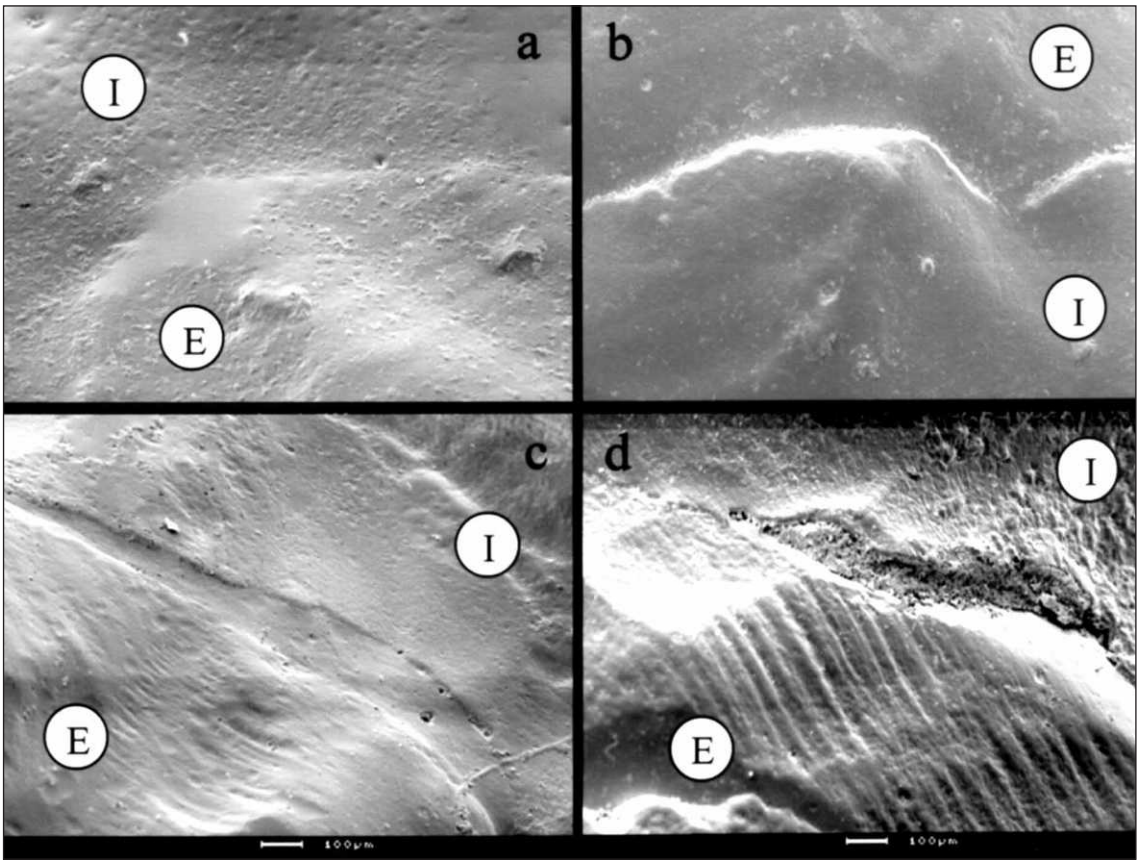


Figure 3. *Four categories of margin quality (I - inlay, E - enamel); a - continuous margin of a composite inlay after the use of two diamonds and MPS gel; b - overhanging margin of a ceramic inlay and c - submargination of a ceramic inlay both after finishing with a 30 µm diamond and a tungsten carbide bur followed by MPS gel; d - marginal imperfection of a ceramic inlay after the use of a 30 µm diamond, a tungsten carbide bur and the diamond polisher.*

six months or longer. In this study, the evaluation was confined to baseline results. The effect of rotary instrumentation on the margins or on restoration surfaces was most evident and clear immediately after finishing and polishing. Marginal imperfections on restorations and surrounding enamel after a longer period of time might be caused by several factors apart from the type of rotary instrumentation. The remaining grooves originating from insufficient polishing might be subject to alteration by attrition during clinical application.

Surface roughness is usually evaluated quantitatively by mechanical or optical profilometry. The application of this method was not possible in this study. Mechanical or optical pick-up systems have a limited vertical dislocation capacity for acquiring roughness data. This depends on the type of stylus: the more precise and greater the resolution, the smaller the vertical dislocation capacity. For example, the vertical capacity for a comparatively crude stylus (FRW 750) with a tip radius of 10 µm is given by ± 750 µm (technical data by Mahr, D-37008 Goettingen, Germany). This means that a vertical difference of more than 1.5 mm within the transverse length cannot be coped with by the pick-up system. With respect to these technical limitations, the anatomical properties of the occlusal surface of posterior teeth will make the application of profilometry very difficult.

The margin analysis demonstrated that none of the methods used for finishing and polishing caused detrimental effects on restoration margins when applied on composite or ceramic inlays. Finishing with a sequence of a 30 µm diamond and a tungsten carbide bur achieved significantly larger amounts of continuous

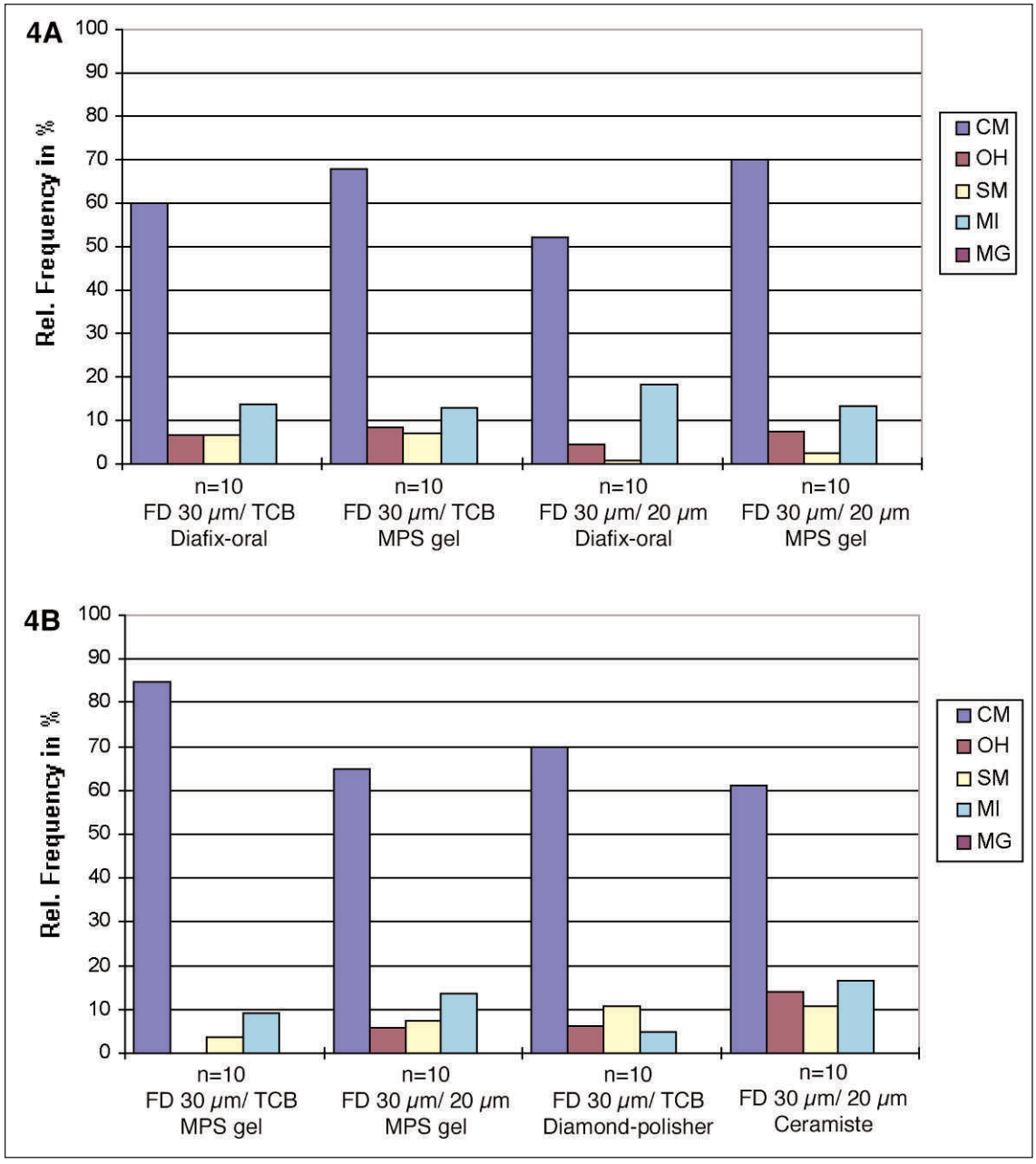


Figure 4. *Portion of margin criteria (Median) of occlusal margins after finishing and polishing according to four different protocols (FD - finishing diamond, TCB - tungsten carbide finishing bur; CM - continuous margin, OH - overhangs, SM - submargination, MI - marginal imperfection, MG - marginal gap)*
A. composite inlays (Tetric)
B. ceramic inlays (IPS Empress)

margins *in vivo*. This could be attributed to the fact that under experimental conditions, tungsten carbide instruments had a greater smoothing effect than finishing diamonds (Jung, 2002; Ward & others, 1995). If the rotary polishing devices, for example, were not able to reach all the surfaces originally instrumented by the rigid finishing instruments, a greater amount of remaining grooves might result from using diamonds compared to tungsten carbide burs.

There were no significant detectable differences between the methods used for finishing and polishing, either with respect to restoration margins or roundness of contours or surface roughness. Several factors might be responsible for this finding. For ethical reasons, only those methods that had proven to be similarly efficient under experimental conditions were selected for the clinical study. Therefore, the detection of significant differences between the methods under *in vivo* conditions was improbable. Furthermore, the accuracy of the qualitative methods used in this study is much smaller than the quantitative evaluation of roughness by profilometry.

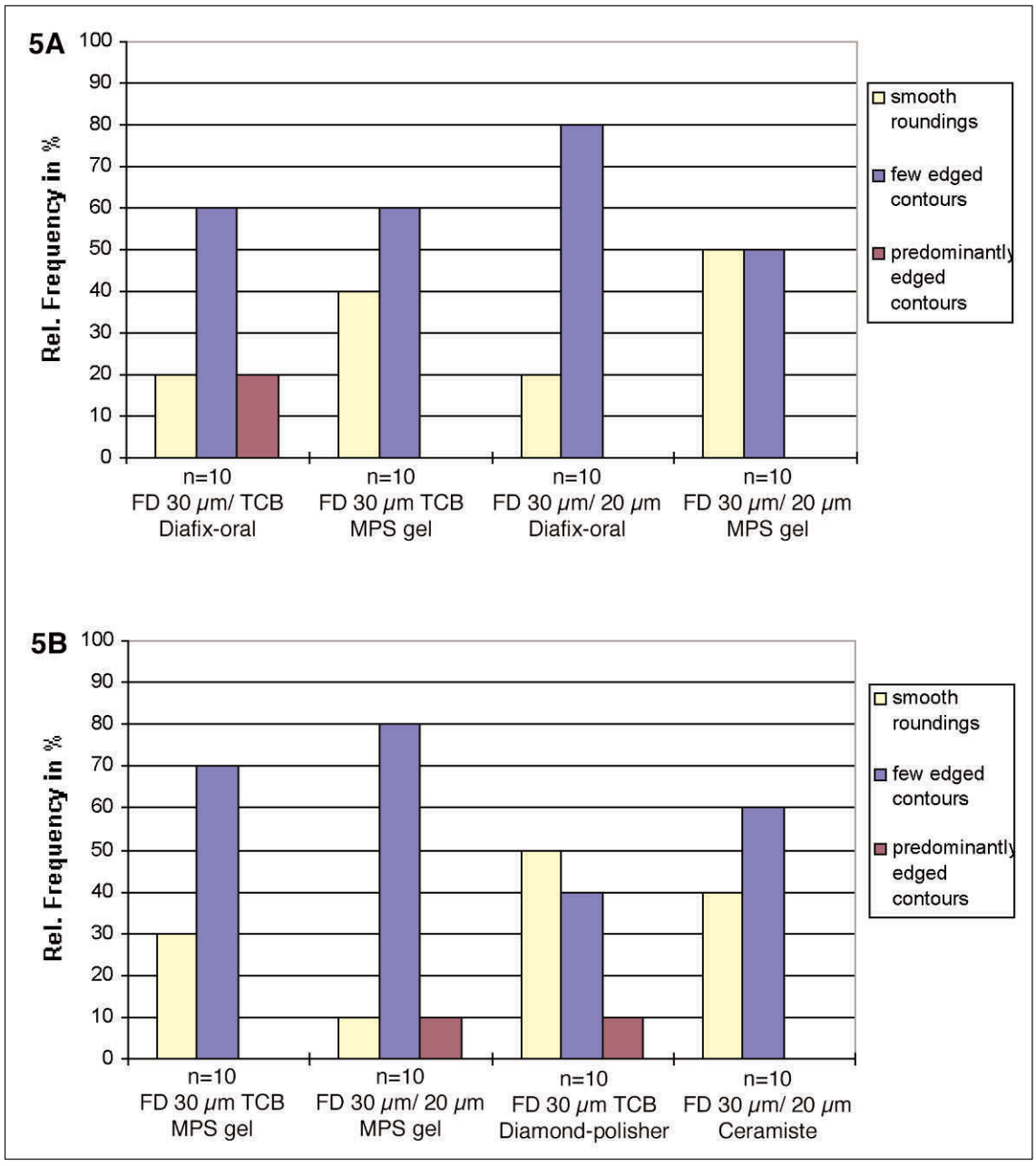


Figure 5. *The portion of three criteria (Mean) characterizing the quality of occlusal contouring after finishing and polishing of Tetric composite inlays (A) and IPS Empress ceramic inlays (B).*

Another difference when comparing the corresponding *in vitro* and in *vivo* results was the behavior of the two materials. Whereas, under experimental conditions, the ceramic surfaces were significantly smoother compared to the composite, no such differences were found under clinical circumstances.

Therefore, the results of this study indicate that the effects of methods for finishing and polishing composites and ceramics *in vitro* were not reproducible *in vivo*.

In accordance with the results of other studies, marginal gap formation after adhesive luting was negligible at baseline (Friedl & others, 1996; Krejci, Krejci & Lutz, 1992; Thonemann & others, 1997; Van Meerbeek & others, 1992). Several studies that evaluate the margin quality of adhesive inlays report on baseline results

similar to those of this study. Sixty to 70% continuous margins, 20% - 30% overhangs and 2% - 5% submargination were found after cementation of different types of composite inlays (Van Meerbeek & others, 1992). Fifty Class II ceramic inlays showed 60% - 75% perfect margins and about 20% - 25% marginal imperfection at baseline (Friedl & others, 1996). In a study on the effect of different luting composites, Noack and others (1993) found a slightly larger portion of continuous margins (80% - 90%), whereas, overhangs and submargination (5% - 10% each) were in a similar dimension compared with this study.

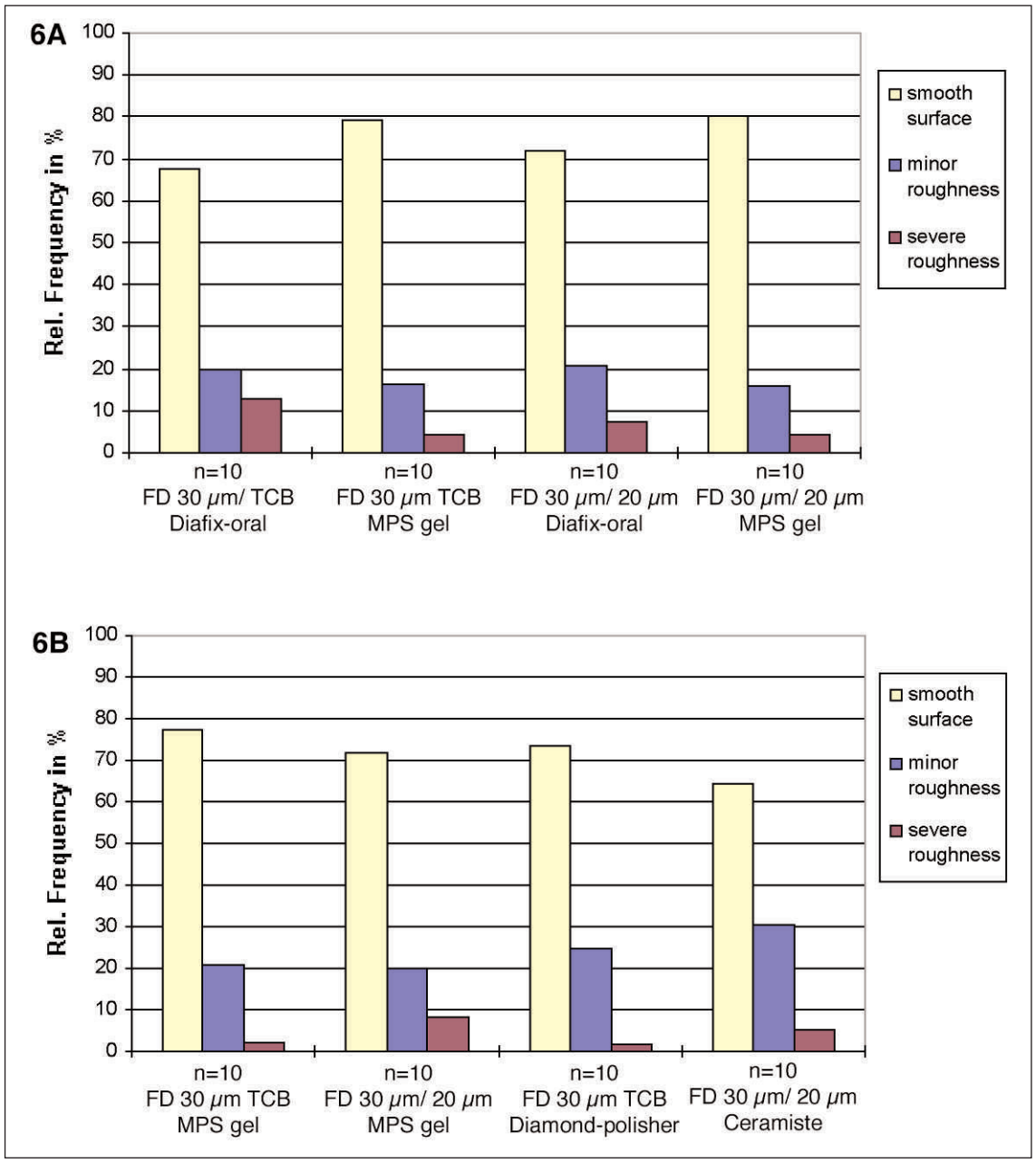


Figure 6. *The portion of three criteria (Mean) describing the surface quality of Tetric composite inlays (A) and IPS Empress ceramic inlays (B) after finishing and polishing according to four different protocols.*

In a clinical study by Krejci and others (1997), the amount of continuous margins ranging >90% was considerably greater; the fact that the restorations evaluated were both direct fillings and indirect inlays might contribute to the differences with respect to the present results.

Few studies have examined the effect of finishing and polishing on adhesive inlays under experimental conditions. In accordance with the current results, finishing composite inlays with a tungsten carbide bur achieved a larger portion of continuous margins (70%) compared to using finishing diamonds (40% perfect margins) (Hannig & others, 1990). The effect of finishing and polishing on the surface of 104 direct Class I composite and ceramic inlays was the subject of another *in-vitro* study (Schmid, Krejci & Lutz, 1991). Rotating stones and rubber polishers turned out to be detrimental. In contrast, finishing diamonds followed by flexible discs caused 88% - 95% perfect margins. This result is questionable, because many authors regard flexible discs to be inefficient with respect to occlusal surfaces (Chen, Chan & Chan, 1988; Karapetian & others, 1996; Tjan & Chan, 1989).

Ashe and others (1996) reported problems when tungsten carbide burs were exclusively applied for finishing

ceramic inserts *in vivo*. Finishing diamonds were less detrimental. These results might be attributable to the low cutting efficacy of tungsten carbide burs, which should therefore not be used exclusively for finishing.

The application of diamond impregnated felt wheels for polishing Cerec inlays was the subject of a study by Kunzelmann and Hickel (1990). The authors observed a selective reduction of the luting composite. In the current study, diamond impregnated felt wheels did not have a comparable effect when applied to composite inlays. The similar physical properties of the luting and inlay composite might be an explanation for these findings.

CONCLUSIONS

None of the instruments used for finishing and polishing caused significant destructive effects on margins and surface of composite and ceramic inlays *in vivo*.

Under clinical conditions, there were no significant differences among the four methods for finishing and polishing composite or ceramic inlays with respect to margin and surface quality.

Using a 30 µm diamond followed by a tungsten carbide finishing bur achieved a significantly larger portion of continuous margins when applied to composite and ceramic inlays compared to a sequence of two diamonds (30 µm and 20 µm).

Because of similar effects on margins and surfaces *in vivo*, those polishing methods that required the smallest number of polishing steps, that is, the diamond impregnated felt wheel with respect to composite inlays and the diamond-polisher with respect to ceramic restorations turned out to be most efficient. Both methods required only one polishing step.

In vivo, there were no differences between composite and ceramic inlays after finishing and polishing with respect to margin and surface quality.

The results of this study demonstrate that those differences that turned out to be of significance under experimental conditions may not be reproducible *in vivo*.

(Received 9 May 2003)

Acknowledgement

The authors express their gratitude to Prof Dr M Noack, University of Koeln, for his support in performing the quantitative margin analysis.

References

Ashe MJ, Tripp GA, Eichmiller FC, George LA & Meiers JC (1996) Surface roughness of glass-ceramic insert—composite restorations: Assessing several polishing techniques *Journal of the American Dental Association* **127(10)** 1495-1500.

Chen RC, Chan DC & Chan KC (1988) A quantitative study of finishing and polishing techniques for a composite *Journal of Prosthetic Dentistry* **59(3)** 292-297.

Friedl KH, Schmalz G, Hiller KA & Saller A (1996) *In-vivo* evaluation of a feldspathic ceramic system: 2-year results *Journal of Dentistry* **24(1-2)** 25-31.

Gemalmaz D, Özcan M & Alkumru HN (2001) A clinical evaluation of ceramic inlays bonded with different luting agents *The Journal of Adhesive Dentistry* **3(3)** 273-283.

Hannig M, Albers HK, Prieshoff T & Weinle S (1990) [SEM study on finishing the joint between inlay and luting composite resin] *Deutsche Zahnärztliche Zeitschrift* **45(10)** 672-675.

Hayashi M, Tsuchitani Y, Kawamura Y, Miura M, Takeshige F & Ebisu S (2000) Eight-year clinical evaluation of fired ceramic inlays *Operative Dentistry* **25(6)** 473-481.

Herrgott AM, Ziemiecki TL & Dennison JB (1989) An evaluation of different composite resin systems finished with various abrasives *Journal of the American Dental Association* **119(6)** 729-732.

Jung M (2002) Finishing and polishing of a hybrid composite and a heat-pressed glass ceramic *Operative Dentistry* **27(2)** 175-183.

Jung M, Baumstieger M & Klimek J (1997) Effectiveness of diamond-impregnated felt wheels for polishing a hybrid composite *Clinical Oral Investigations* **1(2)** 71-76.

Kaplan BA, Goldstein GR, Vijayaraghavan TV & Nelson IK (1996) The effect of three polishing systems on the surface roughness of four hybrid composites: A profilometric and scanning electron microscopy study *Journal of Prosthetic Dentistry* **76(1)** 34-38.

Karapetian VE, Sorg T, Jöckel V & Baumann MA (1996) Comparison of different polishing systems for dental inlay ceramics in *CAD/CIM in Aesthetic Dentistry*. Cerec 10 Year Anniversary Symposium Chicago Quintessence Publishing Co Inc 553-559.

Krejci I, Krejci D & Lutz F (1992) Clinical evaluation of a new pressed glass ceramic inlay material over 1.5 years *Quintessence International* **23(3)** 181-186.

Krejci I, Lutz F, Oddera M & Gschäll H-P (1997) [Amalgam alternatives—evaluation of a clinical concept applied by students] Scientific part of the [Wissenschaftlicher teilder] *Schweizer Monatsschrift für Zahnmedizin* **2(1)** 6-15.

Kunzelmann KH & Hickel R (1990) [Fine polishing of Cerec inlays using diamond polishing systems] *Deutsche Zahnärztliche Zeitschrift* **45(10)** 680-682.

Leirskar J, Henaug T, Thoresen NR, Nordbo H & Von Der Fehr FR (1999) Clinical performance of indirect composite resin inlays/onlays in a dental school: Observations up to 34 months *Acta Odontologica Scandinavia* **57(4)** 216-220.

Molin MK & Karlsson SL (2000) A randomized 5-year clinical evaluation of 3 ceramic inlay systems *International Journal of Prosthodontics* **13(3)** 194-200.

Monaco C, Baldissara P, Dall´Orologio GD & Scotti R (2001) Short-term clinical evaluation of inlay and onlay restorations made with a ceromer *International Journal of Prosthodontics* **14(1)** 81-86.

Noack MJ, Locke LS & Roulet J-F (1993) [Margin quality of porcelain inlays inserted adhesively and by ultrasonic insertion] *Deutsche Zahnärztliche Zeitschrift* **48(11)** 720-723.

Pallesen U & Van Dijken JW (2000) An 8-year evaluation of sintered ceramic and glass ceramic inlays processed by the Cerec CAD/CAM system *European Journal of Oral Sciences* **108(3)** 239-246.

Roulet JF (1987) A materials scientist's view: Assessment of wear and marginal integrity *Quintessence International* **18(8)** 543-552.

Roulet JF, Reich T, Blunck U & Noack M (1989) Quantitative margin analysis in the scanning electron microscope *Scanning Microscopy* **3(1)** 147-159.

Schmid O, Krejci I & Lutz F (1991) [Ausarbeitung von adhäsiven zahnfarbenen Inlays aus Komposit und Keramik] *Schweizer Monatsschrift für Zahnmedizin* **101(2)** 177-184.

Sjögren G, Molin M & Van Dijken JW (1998) A 5-year clinical evaluation of ceramic inlays (Cerec) cemented with a dual-cured or chemically cured resin composite luting agent *Acta Odontologica Scandinavia* **56(5)** 263-267.

Thonemann B, Federlin M, Schmalz G & Schams A (1997) Clinical evaluation of heat-pressed glass-ceramic inlays *in vivo*; 2-year results *Clinical Oral Investigations* **1(1)** 27-34.

Thonemann B, Schmalz G, Brandenstein S & Hiller K-A (1994) [Marginal quality of ceramic inlays with dentin bonding agents *in vitro] Deutsche Zahnärztliche Zeitschrift* **49(10)** 840-844.

Thordrup M, Isidor F & Horsted-Bindslev P (1999) A 3-year study of inlays milled from machinable ceramic blocks representing 2 different inlay systems *Quintessence International* **30(12)** 829-836.

Tjan AH & Chan CA (1989) The polishability of posterior composites *Journal of Prosthetic Dentistry* **61(2)** 138-146.

Van Dijken JW, Hoglund-Aberg C & Olofsson AL (1998) Fired ceramic inlays: A 6-year follow up *Journal of Dentistry* **26(3)** 219-225.

Van Meerbeek B, Inokoshi S, Willems G, Noack MJ, Braem M, Lambrechts P, Roulet JF & Vanherle G (1992) Marginal adaptation of four tooth-coloured inlay systems *in vivo Journal of Dentistry* **20(1)** 18-26.

Ward MT, Tate WH & Powers JM (1995) Surface roughness of opalescent porcelains after polishing *Operative Dentistry* **20(3)** 106-110.

©Operative Dentistry, 2004, **29-2,** 142-149

Laboratory Research

The Nano-Hardness and Elastic Modulus of Carious and Sound Primary Canine Dentin

Y Hosoya • GW Marshall, Jr

Clinical Relevance

The significantly lower nanohardness and elasticity of dentin under the lesion, near the pulp and cervical area, might have a deleterious effect on resin adhesion.

SUMMARY

This study measured the nanohardness and elastic modulus of carious and sound primary canine dentin and compared the values obtained under the lesion and in sound regions of incisal, center and cervical areas, and outer, middle and inner layers. Six extracted or exfoliated primary canines (three with dentin caries on both proximal surfaces and three sound teeth) were mesiodistally sectioned parallel to the long axis of the tooth and polished. The hardness (H), plastic hardness (PH) and Young's modulus (Y) were measured by a nano-indentation tester. Ten indentations at intervals of 10 µm on all regions, areas and layers were made using a load of 1 gf for one second. All indentations were observed using a microscope attached to the tester. All data were statistically analyzed using ANOVA and Scheffe's test at $p<0.05$. For sound teeth, the H, PH and Y values of the inner layer were significantly lower than the outer and middle layers in all areas. The H, PH and Y values of the cervical area were significantly lower than the incisal area in almost all of the outer, middle and inner layers. For carious teeth, the H, PH and Y values of the inner layer were significantly lower than the outer and middle layers in the center area. For the center area, the H, PH and Y values under the lesion were significantly lower than sound teeth in the outer and middle layers. Dentin under the lesion, near the pulp and cervical areas showed significantly lower nanohardness and elasticity.

*Yumiko Hosoya, DDS, PhD, associate professor, Division of Pediatric Dentistry, Department of Developmental and Reconstructive Medicine, Course of Medical and Dental Sciences, Nagasaki University Graduate School of Biomedical Sciences, Nagasaki, Japan

Grayson W Marshall, Jr, DDS, MPH, PhD, professor, Division of Biomaterials and Bioengineering, Department of Preventive and Restorative Dental Science, University of California San Francisco, San Francisco, CA, USA

*Reprint request: 1-7-1, Sakamoto, Nagasaki, 852-8588, Japan; e-mail: hosoya@net.nagasaki-u.ac.jp

INTRODUCTION

Improved resin bonding yields strong bonds to enamel with excellent sealing ability (Retief, 1987). However, the resin-dentin seal is much less reliable. Caries modifies the structure and typical carious lesions contain various zones of the affected layer (Fusayama, Okuse & Hosoda, 1966) that have varying mineral levels and properties. Fusayama (1993) compared the structure and characteristics of three layers of dentin of carious teeth (outer caries dentin, inner carious dentin and normal dentin). Outer caries dentin or the discolored layer

is infected and unremineralizable and should be removed for carious treatment. Inner carious dentin is classified into turbid, transparent and subtransparent layers. These layers are uninfected and remineralizable and should be preserved for caries treatment. Similar zones have been observed in primary teeth with carious lesions (Hosoya & others, 2000; Hosoya, Ono & Marshall, 2002), but only limited work has been done to define the properties of these altered zones. Although permanent tooth dentin has been studied extensively, the microstructure of dentin in primary teeth has received only limited attention. A better understanding of dentin in primary teeth, especially carious primary dentin, is needed to improve dentin bonding methods and make dental restorations more effective and successful.

The hardness and elasticity of fully mineralized permanent dentin have been reported in numerous studies (Craig & Peyton, 1958; Bowen & Rodriguez, 1962; Fusayama & Maeda, 1969; Pashley, Okabe & Parham, 1985; Sano & others, 1994; Kinney & others, 1996; Urabe & others, 2000; Mahoney & others, 2000). Recently, nano-indentation has been used to measure hardness and Young's modulus of dentin on a submicroscopic scale (Urabe & others, 2000; Mahoney & others, 2000). The range in hardness of sound permanent dentin is broad, from 0.2 to 0.8 GPa (1MPa=10.2 kgf/cm^2, 1GPa=101.93675 kgf/mm^2) (Fusayama & Maeda, 1969; Pashley & others, 1985; Sano & others, 1994; Kinney & others, 1996). Young's modulus of sound permanent dentin ranged from about 10 to 20 GPa (Craig & Peyton, 1958; Bowen & Rodriguez, 1962; Lehmann, 1967; Sano & others, 1994; Kinney & others, 1996; Meredith & others, 1996; Xu & others, 1998; Kinney & others, 1999). Few studies of the hardness of primary dentin have been reported (Johnsen, 1994; Mahoney & others, 2000; Hosoya & others, 2000; Hosoya & others, 2002) and Knoop hardness values for sound primary dentin ranged from 35 to 60 KHN (Johnsen, 1994) depending on location within the tooth.

Fusayama and others (1966) and Ogawa and others (1983) reported the Knoop hardness of carious permanent dentin for discolored, transparent and subtransparent layers, and sound dentin and found that all zones, including the transparent zone of the affected layer, were softer than normal dentin. Craig, Gehring and Peyton (1959) also measured the hardness of carious permanent dentin but reported that some zones were harder than normal dentin.

Hosoya and others (2000; 2002) measured the Knoop hardness of carious primary anterior tooth dentin in caries-affected layers including transparent dentin, adjacent sound dentin and dentin regions far from and not related to caries. The results of these reports (Hosoya & others, 2000; Hosoya & others, 2002) showed that primary dentin in areas under lesions and in adjacent regions on the carious side of the teeth had significantly lower hardness than corresponding areas on the sound side. Under the lesion, significantly lower Knoop hardness values were obtained in the region less than 150 μm from the bottom of the cavity and the Ca and P contents at depths less than 100 μm from the bottom of cavity were significantly lower than those at greater distance (Hosoya & others, 2002). Transparent dentin was softer than sound dentin (Hosoya & others, 2000; Hosoya & others, 2002). The reduced mechanical properties of the affected layers of carious dentin suggest that bonding to this weakened structure may be more difficult. This has been reported by several workers for permanent dentin (Nakajima & others, 1995; Nakajima & others, 1999).

The elastic properties of dentin are important for understanding the mechanical properties of calcified tissue and for alterations in the mechanical response due to caries, sclerosis and aging, as well as understanding the effects of adhesive materials to dentin. Mahoney and others (2000) measured the hardness and elastic modulus of sound maxillary primary molar dentin using a nano-indentation tester. However, they used not only sound teeth but also carious teeth as sample teeth and calculated the mixed data. They did not compare the values at different depths or locations of dentin. The elasticity of carious dentin has not been reported for primary carious teeth and only limited studies have been reported for permanent teeth (Marshall & others, 2001).

This study measured the nanohardness and elastic modulus of carious and sound canine dentin and compared the values among areas under the lesions and in sound regions of the incisal, center and cervical areas in the outer, middle and inner layers of each area.

METHODS AND MATERIALS

Sample Teeth

Three sound maxillary primary canines and three mandibular primary canines with caries at the center portion of both proximal surfaces were used for this study. All teeth were extracted or exfoliated by eruption of the succedaneous permanent tooth or extracted as required for orthodontic treatment from Japanese children. The teeth were stored in 4°C physiologic saline solution shortly after extraction or exfoliation. The age of the patients ranged from six years four months to seven years nine months. Informed consent was obtained from the parents and patients in order to collect the teeth. Radiographs were taken to identify the carious areas. The total number of carious lesions was six. In all carious lesions, caries did not extend more than one-fourth the depth of the dentin.

Specimen Preparation

Sound teeth were mesiodistally sectioned parallel to the long axis at the center of the tooth. For carious teeth

with caries on both proximal surfaces, the teeth were longitudinally sectioned through the central part of the largest carious lesion. Sectioning was done using a low-speed saw (Buehler Ltd, Lake Bluff, IL, USA) with a circular diamond blade and copious filtered water.

After sectioning, the specimens were polished on wet silicon carbide paper using grit sizes of 600, 800, 1000 and 1200. Final polishing was carried out on felt cloth using 3, 1, 0.1, 0.3 and 0.05 µm-size aluminum oxide suspensions (Baikowski International Co, Charlotte, NC, USA). Optical photomicrographs of the polished specimens were taken with a microscope (Olympus Co, Tokyo, Japan), and the infected, affected and sound portions of the dentin were identified. The sectioned and polished specimens were stored in 4°C distilled water until measurement and were dried at room temperature for 20 minutes prior to the study.

Nano-Indentation Test

Cyanoacrylate (Konishi Co, Tokyo, Japan) was applied onto small areas of enamel of the specimen, and the specimen was then fixed on a flat glass plate to stabilize its surface and orient the surface parallel to the stage of the nano-indentation tester ENT-1100 (Elionix Co, Tokyo, Japan). ENT-1100 is a depth sensing computer-controlled instrument and has a Berkovich indentor, a three-sided pyramid diamond probe. The instrument was enclosed in an isolation chamber with a temperature controller and placed on an ALD anti-vibration isolator in order to minimize influences of environmental conditions such as room temperature, floor-vibration and noise. The specimen was kept in dry conditions during measurement since this machine can control temperature in the chamber at 26°C but not humidity. The loading control system was powered by electromagnetic force and load ranges from 10 mgf to 100 gf. The position of indentation could be programmed and the indents observed with a CCD camera attached to the tester.

Figure 1 shows a load vs displacement curve from the measurement process. Values of hardness (H), plastic hardness (PH) and Young's modulus (Y) were calculated according to the equations (1), (2) and (3), respectively, following the index of Elionix company that was modified from the method reported by Oliver and Pharr (1992):

$$H=3.7926 \times 10^{-2} \times P_{max}/h_{max}^{2} \quad (1)$$

$$PH=3.7926 \times 10^{-2} \times P_{max}/h_{l}^{2} \quad (2)$$

$$Y=1.81092 \times 10^{-3} \times 1/h_{l} \times dp/dh \quad (3)$$

in which P_{max} is the maximum applied load, h_{max} is the maximum penetration depth, h_1 is the intercept depth and dP/dh is the contact stiffness from the unloading portion of the load vs displacement curve.

Figure 2 shows the names of the areas and layers of the sample teeth for measurement. For sound teeth, the mesial and distal sides were divided into incisal, center and cervical areas, and for the carious teeth, the mesial and distal sides were divided into center and mesial incisal areas. Distal incisal areas were not measured because the small size of the teeth relative to the lesion size suggested that this area would have been altered by caries. The cervical area of the carious teeth was not measured in this study, because this area could not be reliably measured in the carious teeth due to the presence of caries. Then, each area was divided into outer, middle and inner layers. For regions under the lesion, the term "under the lesion" was used instead of the term "outer layer."

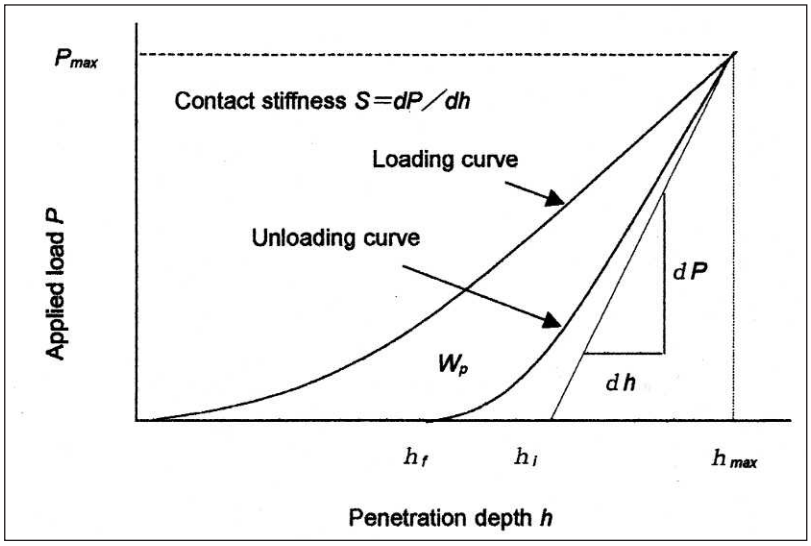


Figure 1. *Load vs displacement curve in the measurement process.*

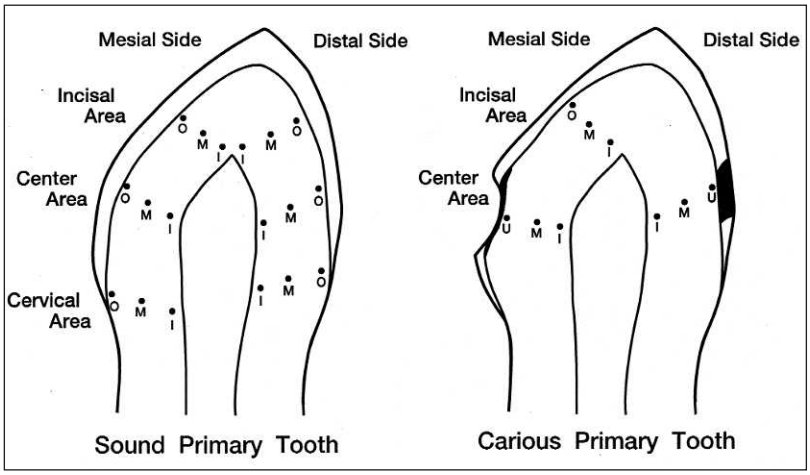


Figure 2. *Names of the areas and layers of the sample teeth in measurement (O: outer layer, M: middle layer, I: inner layer, U: under the lesion layer).*

Ten indentations at intervals of 10 μm in each of the subdivided regions were made perpendicular to the outline of the dentinoenamel junction or DEJ using a load of 1 gf for one second. The positions of the indentations were as follows. The first point of the outer layer was made at 10 μm beneath the DEJ. The first point of the middle layer was made at the center from the DEJ to the pulp chamber wall. The last point of the inner layer was made at the most inner measuring point in primary dentin close to the pulp chamber wall. Indentations were observed using a microscope with a CCD camera attached to the tester under 700x magnification. Some of the indentations were also observed using a scanning electron microscope (SEM) S-3500 (Hitachi Ltd, Tokyo, Japan). For regions under the lesion, measurement was started just internal to the infected and destroyed dentin layer, and the shape of the indentation at the first point was verified by examining the indents with the CCD camera and SEM. Irregular or unclear shaped indentations were removed from the data. Therefore, all of the selected indentations were marked on the intertubular dentin.

All data was statistically analyzed using ANOVA followed by Scheffe's multiple comparison testing and correlation coefficients at $p<0.05$.

RESULTS

First, the means and standard deviations of the values of 10 indentations on each layer were calculated and compared to different areas and layers on the same sample. Tooth to tooth differences existed but regional differences were similar on all samples. Then, the values for all samples were mixed and statistically compared among different areas and layers in this study.

Table 1 shows the hardness, plastic hardness and Young's modulus of sound primary canine dentin. In general, related to the incisal, center and cervical areas, the hardness, plastic hardness and Young's modulus decreased from DEJ to pulp and the values of the inner layer were significantly lower than the outer and middle layers. For cervical areas, the hardness and plastic hardness of the outer layer were significantly higher than the middle and inner layers. Comparing values in the same layer among the different areas, the hardness, plastic hardness and Young's modulus were reduced from incisal to cervical areas and, in general, the values of the cervical area were significantly lower than those of the incisal and center areas.

Table 2 shows the hardness, plastic hardness and Young's modulus of carious primary canine dentin. For the center area or under the decayed area, all values of the inner layer were significantly lower than the outer and middle layers. In the incisal area, which was a sound region of the carious teeth, the values of the outer layer were significantly higher than the middle and inner layers.

Table 3 compares the hardness, plastic hardness and Young's modulus of carious primary canine dentin and sound primary canine dentin. The center area had values under the lesion that were significantly lower than the sound teeth in the outer and middle layers.

Two- or three-way ANOVA and Scheffe's tests indicated that the depth of dentin and caries decay had the most significant influence on the values, and the area of dentin also had a second significant influence on the values.

Figures 3 and 4 show the average hardness and Young's modulus, respectively, for sound and carious primary canine dentin on both the mesial and distal sides of the teeth. The trends for both were similar.

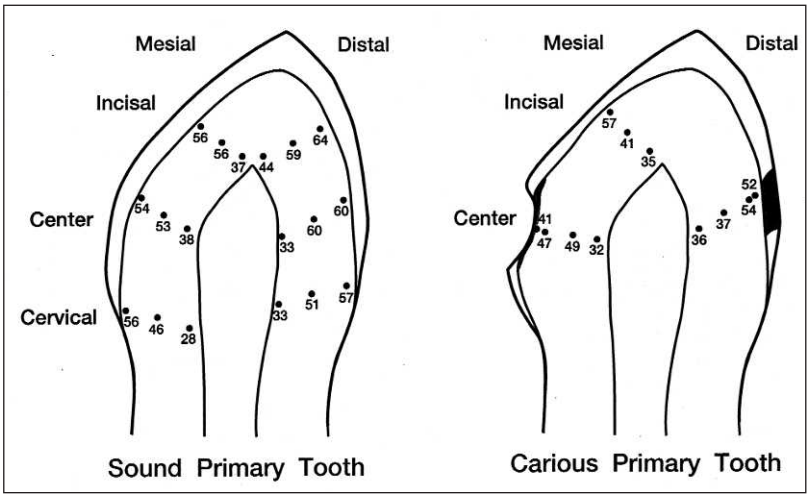


Figure 3. *Average hardness values of sound and carious primary canine dentin.*

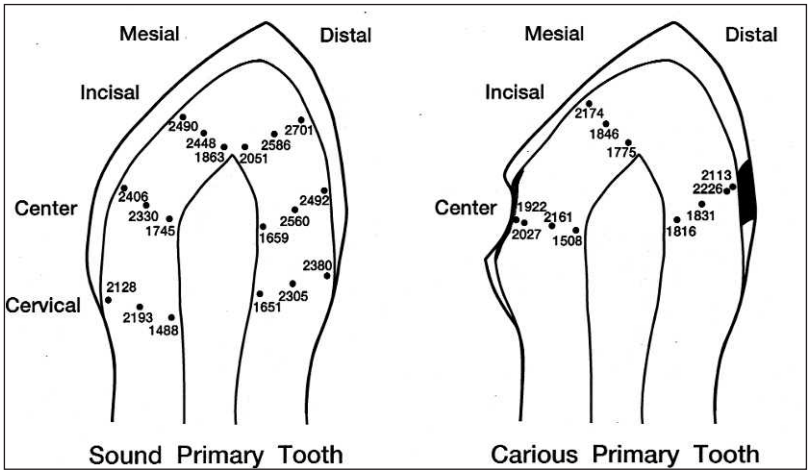


Figure 4. *Average Young's modulus of sound and carious primary canine dentin.*

Table 1: *Hardness, Plastic Hardness and Young's Modulus of Sound Primary Canine Dentin (unit: kgf/mm²)*

Area	Layer	Hardness Mean (SD)	Plastic Hardness Mean (SD)	Young's Modulus Mean (SD)	Number of Measurements
Incisal	Outer	60.0 (8.0)	71.5 (12.6)	2597 (367)	59
	Middle	57.3 (13.9)	74.3 (19.7)	2517 (423)	60
	Inner	40.6 (10.7)	50.6 (14.8)	1957 (364)	60
Center	Outer	57.0 (11.3)	71.6 (15.8)	2447 (393)	60
	Middle	56.8 (12.6)	71.6 (18.0)	2445 (379)	58
	Inner	35.4 (10.4)	44.3 (14.5)	1702 (347)	60
Cervical	Outer	56.8 (6.7)	76.5 (11.7)	2254 (341)	60
	Middle	48.7 (12.4)	60.8 (18.8)	2249 (312)	60
	Inner	30.6 (9.4)	36.6 (13.4)	1569 (263)	60

Vertical line: no significant difference at $p<0.05$

Table 2: *Hardness, Plastic Hardness and Young's Modulus of Carious Primary Dentin (unit: kgf/mm²)*

Area	Layer	Hardness Mean (SD)	Plastic Hardness Mean (SD)	Young's Modulus Mean (SD)	Number of Measurements
Center	Under Lesion	46.9 (19.5)	57.5 (25.0)	2023(618)	59
	Middle	42.9 (17.7)	53.5 (23.5)	1996 (700)	60
	Inner	33.6 (12.0)	41.0 (15.8)	1662 (498)	60
Incisal	Outer	57.1 (15.1)	77.2 (20.7)	2174 (593)	29
	Middle	40.7 (21.0)	52.0 (30.2)	1846 (596)	30
	Inner	34.8 (11.2)	42.0 (14.7)	1775 (429)	30

Vertical line: no significant difference at $p<0.05$

Table 3: *Hardness, Plastic Hardness, and Young's Modulus of Carious and Sound Primay Dentin (unit: kgf/mm²)*

Layer	Tooth	Area	Hardness Mean (SD)	Plastic Hardness Mean (SD)	Young's Modulus Mean (SD)	Number of Measurements
Under Lesion	Carious	Center	46.9 (19.5)	57.5 (25.0)	2023 (618)	59
Outer	Carious	Incisal	57.1 (15.1)	77.2 (20.7)	2174 (593)	29
Outer	Sound	Center	57.0 (11.3)	71.6 (15.8)	2447 (393)	60
Middle	Carious	Center	42.9 (17.7)	53.5 (23.5)	1996 (700)	60
Middle	Carious	Incisal	40.7 (21.0)	52.0 (30.2)	1846 (596)	30
Middle	Sound	Center	56.8 (12.6)	71.6 (18.0)	2445 (379)	58
Inner	Carious	Center	33.6 (12.0)	41.0 (15.8)	1662 (498)	60
Inner	Carious	Incisal	34.8 (11.2)	42.0 (14.7)	1776 (429)	30
Inner	Sound	Center	35.4 (10.4)	44.3 (14.5)	1702 (347)	60

Vertical line: no significant difference at $p<0.05$

Under the lesion, not only the average values but also the values at the first measuring point located in the discolored layer were plotted on the figures. Values under the lesion and in the middle layers under the decayed areas or center area of the carious teeth were significantly lower than either the outer or middle layers at the center areas in the sound teeth.

Hardness and Young's modulus showed significant correlations (corr = 0.932, $p<0.0001$).

DISCUSSION

The nano-indentation technique has several advantages for determining hardness over conventional microhardness methods such as Vickers and Knoop hardness. This technique has the ability to produce small indentations under small loads and can measure both the hardness and elastic modulus of materials. Previous reports have suggested wide variations in the basic mechanical properties of dentin. Some of this variation may result from using techniques such as microhardness, which yield averaged values that include contributions from the tubules, peritubular dentin and intertubular dentin. Since the quality of each dentin component might vary with location, this could contribute to the wide range of values. However, work by Kinney and others (1996) indicated that much

of the variation in permanent teeth could result from differences in intertubular dentin rather than peritubular dentin. In this work, we used a nanoindentation technique that allowed measurement of the intertubular dentin alone.

Sound Teeth

In this study, the hardness values of sound primary canine dentin ranged from 30 to 60 kgf/mm^2 and plastic hardness ranged from 37 to 77 kgf/mm^2 (Table 1, Figure 3). These values were lower than that for primary molars (0.92 GPa or 94 kgf/mm^2) as reported by Mahoney and others (2000) but were in agreement with previous studies of sound permanent dentin (Fusayama & Maeda, 1969; Pashley & others, 1985; Sano & others, 1994; Kinney & others, 1996). Hardness is calculated by maximum penetration depth that includes both plastic and elastic deformation of dentin. Plastic hardness is calculated based only on the plastic deformation of dentin and corresponds to Vicker's hardness. In this study, variations in hardness and plastic hardness were almost the same for all layers and areas. Young's modulus of dentin for sound primary canines ranged from about 1600 to 2600 kgf/mm^2 (Table 1, Figure 4). These values were in agreement with a previous study of primary molar dentin (19.89 GPa or 2029 kgf/mm^2) (Mahoney & others, 2000) and sound permanent dentin (Craig & Peyton, 1958; Bowen & Rodriguez, 1962; Lehmann, 1967; Sano & others, 1994; Kinney & others, 1996; Meredith & others, 1996; Xu & others, 1998; Kinney & others, 1999).

No report using a nano-indentation technique has compared the hardness and elasticity among the depths and areas of primary dentin. Meredith and others (1996) and Hosoya and others (2000; 2002) reported that the hardness of dentin decreased with distance from the dentinoenamel junction, while Pashley and others (1985) reported a highly significant inverse correlation in permanent dentin between dentin microhardness and tubule numerical density that increases with depth. However, Kinney and others (1996) reported that most of the decrease in dentin hardness upon approaching the pulp chamber could be attributed to changes in the hardness of intertubular dentin, not just the increase in the number of tubules.

In this study, for all areas, hardness, plastic hardness and Young's modulus of the inner layer were significantly lower than that of the outer and middle layers (Table 1). These results for the hardness and plastic hardness agreed with previous studies (Craig & Peyton, 1958; Bowen & Rodriguez, 1962; Lehmann, 1967; Sano & others, 1994; Kinney & others, 1996; Meredith & others, 1996; Kinney & others, 1999), but the result of the Young's modulus was in disagreement with suggestions by Kinney and others (1999). They utilized atomic-force microscope (AFM) based nanoindentation measurements and suggested that Young's modulus was dominated by the properties of intertubular dentin and could show a slight increase from outer to inner dentin. However, the current study showed significant correlations among the values of hardness and Young's modulus, which were in agreement with the previous report for primary molars (Mahoney & others, 2000).

In this study, there was no significant difference in hardness, plastic hardness and Young's modulus between the outer and middle layers of all areas, except for the hardness and plastic hardness of the cervical area. Since the outer layer of this study was positioned 10-110 μm beneath the DEJ, the finding that there was no significant hardness difference between the outer and middle layers might be due to the influence of the mantle dentin.

Carious Teeth

Craig and others (1959) reported that permanent dentin surrounding a caries lesion had a hardness of 10 KHN greater than sound dentin; the hardness at the center of the lesion was significantly lower and transparent dentin was 10 KHN harder than the adjacent area. Fusayama and others (1966) and Ogawa and others (1983) reported that Knoop hardness for the discolored layer of carious permanent dentin ranged from 20 to 27 KHN, the transparent layer from 27 to 48 KHN, the subtransparent layer from 48 to 68 KHN, and sound dentin varied from 21 to 68 KHN. In our previous study (Hosoya & others, 2002), the average Knoop hardness values of primary canine dentin under the lesion ranged from 31 to 43 KHN in the outer layer, 35 to 44 KHN in the middle layer and 30 to 43 KHN in the inner layer, respectively. However, values less than 150 μm from the bottom of cavity were about 29 KHN and were lower than the region 250 μm away from the cavity. The hardness of many layers and areas of the carious side of carious primary canines, even in apparently unaffected dentin, were significantly lower than the sound side of carious primary canines, suggesting that they were affected by caries. In our previous study on carious primary anterior teeth (Hosoya & others, 2000), the transparent layer had significantly higher hardness than the more superficial decayed layer, but it was not significantly different from adjacent areas or areas to the side of the lesion. In another former study (Hosoya & others, 2002), there was no significant difference in hardness between transparent dentin and non-transparent dentin in the same areas and layers. Marshall and others (2001) reported that the hardness and elastic modulus of intertubular dentin decreased slightly or was unchanged in the transparent dentin of permanent teeth.

In the incisal area that was sound in carious teeth, the hardness, plastic hardness and Young's modulus of

the outer layer were significantly higher than the middle and inner layers. However, in the center area that was under the decayed area, there was no significant difference in hardness, plastic hardness and Young's modulus under the lesion (outer) and middle layers (Table 2). All mechanical values under the lesion were significantly lower than the corresponding regions of sound teeth (Table 3), although caries did not extend more than one-fourth the depth of the dentin in the teeth used for this study. Therefore, the results of this study indicate that not only in the obviously affected layer but also in the areas adjacent to the lesion not obviously affected by caries, the dentin was softer and had a lower elastic modulus than sound dentin (Figures 3 and 4). In this study, the lesions were small and only one linear measurement was done under the carious area. Therefore, the values of the transparent dentin could not be statistically analyzed. However, no significant difference was obtained between the values of the first measuring point under the lesion, which was positioned 10 μm from the bottom of cavity and the discolored dentin and the average values under the lesion, which included all measurements 10-110 μm from the bottom of cavity and included not only discolored dentin but also transparent dentin (Figures 3 and 4). This further suggests that transparent dentin may not be sclerotic as noted by other studies (Fusayama & others, 1966; Ogawa and others, 1983; Hosoya and others, 2000; Marshall and others, 2001).

The results of this study showed that hardness and elastic modulus of dentin differed with intratooth location and decreased with distance from the DEJ in primary canine dentin (Tables 1-3, Figures 3 and 4). Hardness and elastic modulus of dentin might also differ with tooth type and environmental factors during the time of tooth formation and mineralization. In this study, all the sound teeth were maxillary primary canines but all the carious teeth were mandibular primary canines. In addition, the position of the carious lesion was almost at the center area of the proximal surfaces but was not the same among the specimens. The depth from the DEJ to the first measuring point under the lesion differed among the carious teeth and differed between the sound and carious teeth. These differences, due to varying lesion shapes, caused considerable variation among the specimens but, in general, both dentin under the lesion and apparently normal dentin adjacent to the lesion had reduced hardness and elastic modulus compared to sound dentin. None of the areas associated with carious teeth had increased values, thus, there was no evidence of sclerotic dentin in these lesions.

Although the use of nano-indentation permits smaller areas than microhardness to be measured, both the microhardness and nano-indentation equipment used for this study were carried out on dried tissues. When demineralized dentin is dried, it collapses (Marshall & others, 1998), and indentations on the collapsed layer could be influenced by the underlying mineralized tissue. Improved methods such as an AFM-based indentation, which allows submicroscopic indentations of hydrated tissues, may be needed to improve accuracy of the values of various altered layers of carious dentin (Balooch & others, 1998; Marshall & others, 2001).

This study and our previous work (Hosoya & others, 2000; Hosoya & others, 2002) indicate that lower hardness and elastic modulus values were obtained under the caries region and also in the region adjacent to the caries that was apparently not affected by the lesion. This could have a deleterious effect on resin adhesion to these regions. After etching, the hardness might be lower or the depth of demineralization might be increased so that bonding to these areas might require specific etching treatments that have not yet been defined. Previous reports (Nor & others, 1997; Olmez & others, 1998; Hosoya & others, 2001) suggested that primary dentin is more susceptible to acid or some chemical conditioning treatments and, therefore, shorter application times for the dentin conditioner in primary dentin may be appropriate. Further study is required to understand the precise mechanism of adhesion between carious dentin and resinous materials and whether different treatments are needed to optimize bonding for primary and permanent dentin.

CONCLUSIONS

1. Hardness and elastic modulus of sound primary canine dentin were consistent with prior reported results for permanent dentin.
2. Hardness and elastic modulus for primary canine teeth with carious lesions showed markedly lower mechanical properties than sound primary dentin. No areas with increased mechanical properties were observed, thus, there was no evidence of sclerotic dentin associated with primary canine caries.
3. Apparently unaffected dentin in areas adjacent to carious lesions had reduced mechanical properties compared to primary dentin in sound teeth.

Acknowledgements

The authors acknowledge Mr Hideo Suzuki and Mr Takahiko Uematsu (Elionix Co) for their technical assistance in the operation of the nanoindentation tester. This study was partially supported by the Japanese Ministry of Education, Science, Sport and Culture grant 11672053 and 14571955.

(Received 30 January 2003)

References

Balooch M, Wu-Magidi IC, Lundkvist AS, Marshall SJ, Marshall GW Jr, Seikhaus WJ & Kinney JH (1998) Viscoelastic properties of demineralized human dentin in measured water with Atomic force microscope (AFM) -based indentation *Journal of Biomedical Materials Research* **40(4)** 539-544.

Bowen RL & Rodriguez MM (1962) Tensile and modulus of elasticity of tooth structure and several restorative materials *Journal of the American Dental Association* **64(7)** 378-387.

Craig RG & Peyton FA (1958) Elastic and mechanical properties of human dentin *Journal of Dental Research* **37(4)** 710-718.

Craig RG, Gehring PE & Peyton FA (1959) Relation of structure to the microhardness of human dentin *Journal of Dental Research* **38(3)** 624-630.

Fusayama T, Okuse K & Hosoda H (1966) Relationship between hardness, discoloration, and microbial invasion in caries dentin *Journal of Dental Research* **45(4)** 1033-1046.

Fusayama T & Maeda T (1969) Effect of pulpectomy on dentin hardness *Journal of Dental Research* **48(3)** 452-460.

Fusayama T (1993) New concepts in the pathology and treatment of dental caries. A simple pain-free adhesive restorative system by minimal reduction and total etching Ishiyaku Euro America Inc, St Louis, MO p 1-16.

Hosoya Y, Marshall SJ, Watanabe LG & Marshall GW Jr (2000) Microhardness of carious deciduous dentin *Operative Dentistry* **25(2)** 81-89.

Hosoya Y, Kawashita Y, Marshall GW Jr & Goto G (2001) Influence of Carisolv™ for resin adhesion to sound human primary dentin and young permanent dentin *Journal of Dentistry* **29(3)** 163-171.

Hosoya Y, Ono T & Marshall GW Jr (2002) Microhardness of carious primary canine dentin *Pediatric Dental Journal* **12(1)** 91-98.

Johnsen DC (1994) *Comparison of Primary and Permanent Teeth Oral Development and Histology* 2nd ed JK Avery, New York: Thieme Medical Publishers p 287.

Kinney JH, Balooch M, Marshall SJ, Marshall GW Jr & Weihs TP (1996) Hardness and Young's modulus of human peritubular and intertubular dentin *Archives of Oral Biology* **41(1)** 9-13.

Kinney JH, Balooch M, Marshall GW Jr & Marshall SJ (1999) A micromechanics model of the elastic properties of human dentine *Archives of Oral Biology* **44(10)** 813-822.

Lehmann ML (1967) Tensile strength of human dentin *Journal of Dental Research* **46(1)** 197-201.

Mahoney E, Holt A, Swain M & Kilpatrick N (2000) The hardness and modulus of elasticity of primary molar teeth: An ultra-micro-indentation study *Journal of Dentistry* **28(8)** 589-594.

Marshall GW Jr, Wu-Magidi IC, Watanabe LG, Inai N, Balooch M, Kinney JH & Marshall SJ (1998) Effect of citric acid concentration on dentin demineralization, dehydration and rehydration: An atomic force microscopy study *Journal of Biomedical Materials Research* **42(2)** 500-507.

Marshall GW Jr, Habelitz S, Gallagher R, Balooch M & Marshall SJ (2001) Nanomechanical properties of hydrated carious human dentin *Journal of Dental Research* **80(8)** 1768-1771.

Meredith N, Sherriff M, Setchell DJ & Swanson SA (1996) Measurement of the microhardness and Young's modulus of human enamel and dentine using an indentation technique *Archives of Oral Biology* **41(6)** 539-545.

Nakajima M, Sano H, Burrow MF, Tagami J, Yoshiyama E, 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.

Nakajima M, Ogata M, Okuda M, Tagami J, Sano H & Pashley DH (1999) Bonding to caries-affected dentin using self-etching primers *American Journal of Dentistry* **12(6)** 309-314.

Nor JE, Feigal RJ, Dennison JB & Edwards CA (1997) Dentin bonding: SEM comparison of the dentin surface in primary and permanent teeth *Pediatric Dentistry* **19(4)** 246-252.

Ogawa K, Yamashita Y, Ichijo T & Fusayama T (1983) The ultrastructure and hardness of the transparent layer of human carious dentin *Journal of Dental Research* **62(1)** 7-10.

Olmez A, Oztas N, Basak F & Erdal S (1998) Comparison of the resin-dentin interface in primary and permanent teeth *Journal of Clinical Pediatric Dentistry* **22(4)** 293-298.

Oliver WC & Pharr GW (1992) An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments *Journal of Materials Research* **7(6)** 1564-1583.

Pashley DH, Okabe A & Parham P (1985) The relationship between dentin microhardness and tubule density *Endodontics and Dental Traumatology* **1(5)** 176-179.

Retief DH (1987) Are adhesive techniques sufficient to prevent microleakage? *Operative Dentistry* **12(4)** 140-145.

Sano H, Ciucchi B, Matthews WG & Pashley DH (1994) Tensile properties of mineralized and demineralized human and bovine dentin *Journal of Dental Research* **73(6)** 1205-1211.

Urabe I, Nakajima M, Sano H & Tagami J (2000) Physical properties of the dentin-enamel junction region *American Journal of Dentistry* **13(3)** 129-135.

Xu HHK, Smith DT, Jahanmir S, Romberg E, Kelly JR, Thompson VP & Rekow ED (1998) Indentation damage and mechanical properties of human enamel and dentin *Journal of Dental Research* **77** 472-480.

©Operative Dentistry, 2004, **29-2**, 150-156

Effect of Thermal and Mechanical Load Cycling on Microtensile Bond Strength of a Total-Etch Adhesive System

AKB Bedran-de-Castro • PNR Pereira
LAF Pimenta • JY Thompson

Clinical Relevance

The combination of thermal and mechanical cycling adversely affected bond strengths, resulting in a possible simulation of the oral stresses incurred during the life span of a restoration.

SUMMARY

To evaluate the effect of thermal and mechanical cycles on dentin bond strength to cervical margins of Class II restorations, 80 box-type Class II cavities were prepared on the surfaces of bovine incisors. The cavities were restored with Single Bond (3M-ESPE) and Z-250 composite (3M-ESPE) according to manufacturer's instructions. The incisors were divided into four groups: G1-Control, G2- Thermal cycling (2,000 cycles, 5°C - 55°C), G3- Mechanical cycling (100,000 cycles; 50N) and G4- Thermal and mechanical cycling (2,000 cycles 5°C-55°C/100,000 cycles; 50N). The restorations were sectioned perpendicular to the cervical bonded interface into 0.7 ± 0.2 mm-thick slabs. The slabs were further trimmed at the interface to 1.4 ± 0.2 mm with a fine diamond bur to produce a cross-sectional surface area of 1 mm². All specimens were then subjected to microtensile bond testing. Means and standard deviations were expressed in MPa. The bond strength data were analyzed by one-way ANOVA and Fisher's PLSD test ($p<0.05$). Fracture mode analysis was performed using SEM. Bond strengths were significantly lower when thermal and mechanical cycling were performed [G4-2.41 (8.57)] when compared to the other groups [G1-28.15 (14.03); G2-27.60 (10.14); G3- 27.59 (8.67)]. No differences were observed among Groups 1, 2 and 3. Interfacial fracture of the control (G1) and thermocycling (G2) groups mainly occurred between the deepest portion of the adhesive resin and the top layer of the demineralized dentin (Interphase). Mixed failure was predominant and

*Ana Karina B Bedran-de-Castro, DDS, MS, PhD, research fellow, Department of Restorative Dentistry, Piracicaba School of Dentistry/Unicamp–Piracicaba-SP-Brazil and Department of Operative Dentistry, School of Dentistry, University of North Carolina-Chapel Hill, NC, USA

Patricia NR Pereira, DDS, PhD, assistant professor, Department of Operative Dentistry, School of Dentistry, University of North Carolina-Chapel Hill, NC, USA

Luiz Andre F Pimenta, DDS, MS, PhD, associate professor, Piracicaba School of Dentistry—Unicamp–Department of Restorative Dentistry, Piracicaba-SP-Brazil

Jeffrey Y Thompson, BS, PhD, associate professor, Department of Operative Dentistry, School of Dentistry, University of North Carolina-Chapel Hill, NC, USA

*Reprint request: 306 Brauer Hall CB#7450, Chapel Hill, NC 27516-7450, USA; e-mail: anakarina_bedran@dentistry.unc.edu

increased when mechanical cycling was applied (G3 and G4).

INTRODUCTION

The control of composite polymerization shrinkage is the first step to achieving and maintaining the integrity of the restoration interface. Polymerization shrinkage results in stress at the dentin/restoration interface, allows for non-adaptation of the material (Jorgensen & others, 1985) and, consequently, accelerates marginal leakage and deterioration of the restoration (Abdalla & Davidson, 1993; Alani & Toh, 1997; Miyazaki & others, 1998). Deterioration of the restoration could subsequently occur due to chemical, thermal and mechanical load stresses (Jorgensen & others, 1985; Abdalla & Davidson, 1993; Abdalla & Davidson, 1996; da cunha Mello & others, 1997).

The establishment of *in vitro* methodologies using different types of stresses present in the oral environment is important since the constant and rapid evolution of the adhesive materials does not allow for long-term clinical trials. The use of thermal and mechanical stresses is used in *in vitro* studies in order to mimic the natural aging process of the restoration. Studies that evaluate microleakage have shown conflicting results regarding the effectiveness of both mechanical (Darbyshire, Messer & Douglas, 1988; Abdalla & Davidson, 1993; Prati & others, 1994; Abdalla & Davidson, 1996; Hakimeh & others, 2000; Nara & others, 2002) and thermal stresses (Darbyshire & others, 1988; Chan & Glyn-Jones, 1994; Hakimeh & others, 2000;) and very limited bond strength studies have been conducted that associate these stresses (Miyazaki & others, 1998; Ausiello & others, 1999; Nikaido & others, 2002a; Nikaido & others, 2002b).

With the microtensile bonding test (Sano & others, 1994), measurement of bond strength to small surface areas (0.5-1.0 mm^2) has become possible. It permits the testing of irregular surfaces and cavities that are more clinically relevant than flat surfaces but which were still not possible with conventional shear and tensile bond tests. The benefit of possibly evaluating small areas such as cervical, gingival and axial walls and the pulp floor of Class I, II and V preparations is enormous and closer to the clinical situation since clinicians usually do not bond to flat surfaces with a very small C-factor. C-factor is the ratio of the bonded to the unbonded or free surface area (Feilzer, de Gee & Davidson, 1987). Box-like Class II cavities can have four bonded walls and two unbonded surfaces, giving them a C-factor of three if all the walls have the same surface area. In flat dentin surface testing, since there are no walls to bond, the C-factor value (one bonded/five unbonded = 0.2) is very low and may overestimate the bond strengths that can be achieved in complex cavities prepared and restored under clinically relevant conditions (Bouillaguet & others, 2001).

This study evaluated the effect of mechanical and thermal cycling on microtensile bond strength (μTBS) and failure mode pattern at the cervical margins of Class II slot restorations. The null hypothesis tested was that thermal and mechanical cycling would not affect bond strength and failure modes.

METHODS AND MATERIALS

Eighty bovine incisors were selected, cleaned of debris with curettes and pumice paste in slow speed and stored in a 0.1% sodium azide saline solution. Incisal surfaces were cut 4 mm above the cementum-enamel junction under water irrigation.

Class II slot preparations were prepared on the mesial surface following the dimensions: 3 mm wide, 5 mm high (starting at the marginal ridge and with the gingival margins in dentin) and 1.5 mm deep toward the pulp chamber. All cavities were prepared using carbide burs (#245 KG Sorensen, Barueri, SP, Brazil) mounted in a high-speed handpiece under water irrigation. The burs were replaced after every five preparations.

The cavities were restored with Single Bond adhesive system (3M-ESPE Dental Products, St Paul, MN, USA) and resin composite Z-250 (3M-ESPE Dental Products). The adhesive system was applied according to manufacturers' instructions: acid etch for 15 seconds, rinse for 15 seconds, blot dry with a cotton pellet, apply two consecutive coats of the adhesive, lightly air dry and light-cure for 10 seconds. The preparation was filled with a micro-hybrid resin composite Z-250 (3M-ESPE Dental Products) in two horizontal increments and light cured for 40 seconds each. A 1-mm overfill was left on the occlusal surface to enable mechanical load cycling on the restoration only. During all restorative procedures, the light intensity (Optilux 501, Demetron/Kerr Corp, Orange, CA, USA) was measured periodically by a radiometer (Demetron/Kerr Corp) and ranged from 520 to 560 mW/cm^2. After the restorative procedure, the specimens were stored in distilled/deionized water at 37°C for 24 hours. They were then finished and polished with Al_2O_3 abrasive discs (Sof-Lex Pop-on/, 3M ESPE). The teeth were then randomly divided into four groups:

G1= control group (no thermal and mechanical load cycling)

G2= thermal cycling only (2,000 cycles, 5°C to 55°C)

G3= mechanical load cycling only (100,000 cycles/load=50N)

G4=thermal cycling (2,000 cycles, 5°C to 55°C) and mechanical cycling (100,000 cycles/load=50N)

The specimens subjected to mechanical load cycling had part of their roots embedded in epoxy resin (Buehler Ltd, Lake Bluff, IL, USA) in order to obtain a flat occlusal surface perpendicular to the long axis of the tooth.

Thermal Cycling and Mechanical Load Cycling Procedure

Specimens from Groups G2 and G4 were subjected to 2,000 cycles in a thermocycling apparatus (MCT2–AMM–2, Sao Paulo, SP, Brazil) with two baths at 5 ± 2°C and 55 ± 2°C each with a dwell time of 60 seconds and a transfer time of seven seconds between each bath (Nikaido & others, 2002b).

Specimens from Groups G3 and G4 were subjected to mechanical load cycling. The cyclic mechanical loading device used was a Leinfelder Wear Test Apparatus (custom made by Dentsply/Caulk Technical Research, Milford, DE, USA) modified for loading test. The apparatus consisted of four stainless pistons with a polyacetal cylindrical tip attached to the end (Figure 1). The polyacetal tips were placed in contact with the restoration. The loading device delivered an intermittent axial force of 50 N at two cycles/second, totaling 100,000 cycles (Hakimeh & others, 2000) (Figure 2-A). For Group G4, thermal cycling was performed first followed by the mechanical cycling.

Microtensile Bonding Test

After thermal and mechanical load cycling, a 3-mm thick resin block was built on the outermost surface of the restoration in order to produce grips for the microtensile bond test. The restorations were sectioned perpendicular to the cervical bonded interface of each tooth into 0.7 ± 0.2-mm thick slabs (n=2 per restoration) with a slow speed diamond wafering blade (Buehler-Series 15LC Diamond) and constant water coolant (Figure 1-B). The composite/dentin interface was further trimmed at the interface to 1.4 ± 0.2 mm with a fine diamond bur to produce a cross-sectional surface area of 1 mm^2 (Figure 2-B). All specimens were then glued with Zapit (DVA, Corona, CA, USA) to a Ciucchi's jig that was mounted on a universal testing machine (EZ Test, Shimazu Co, Kyoto, Japan) and subjected to microtensile bond testing at a crosshead speed of 1 mm/minute. Means and standard deviations were calculated and expressed in MPa. Statistical analysis was performed using ANOVA and Fisher's PLSD test ($p<0.05$).

Fracture Mode Analysis

For fracture mode analysis, specimens were stored in 10% neutral buffered formalin solution for at least eight hours after debonding. All specimens were then mounted on stubs, gold sputter coated (Polaron E-5200 Energy Beam Sciences, Agawan, MA, USA) and examined in a scanning electron microscope (model 6500, JEOL Corp, Peabody, MA, USA). Fracture modes were classified as failure between the deepest portion of the adhesive resin and the top layer of the demineralized dentin [Interphase] (Nakabayashi & Pashley, 1998), cohesive failure in composite, cohesive failure in adhesive and mixed failure (association of two or more failures). Fractures were calculated according to the percentage present in each specimen.

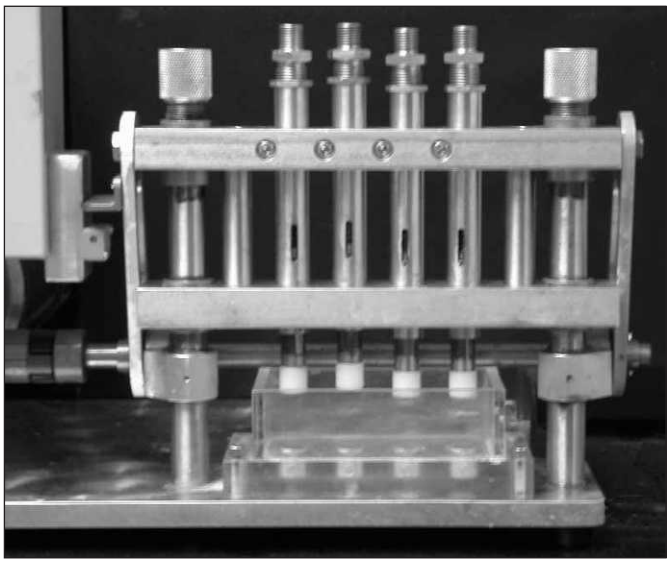


Figure 1. *Illustration of the Leinfelder Wear Test Apparatus used for mechanical load cycling.*

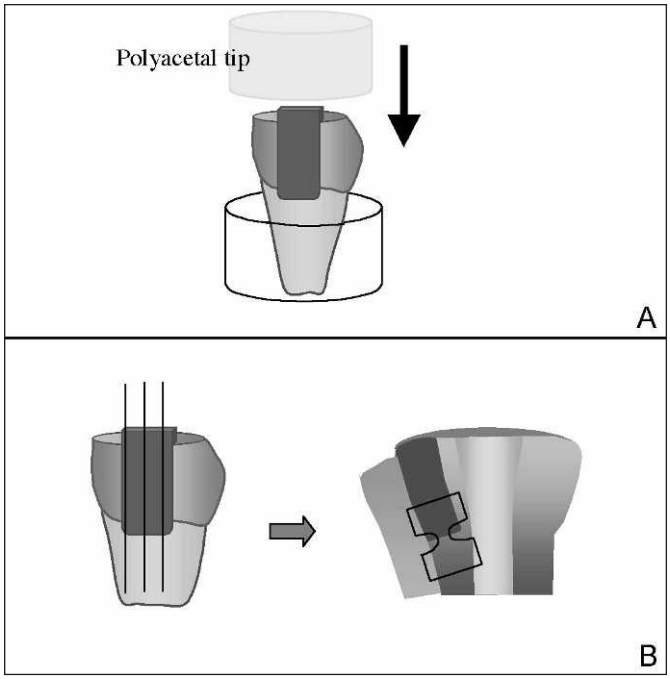


Figure 2. *A- Illustration of the load application on the occlusal surface. B- Illustration of the restoration section and slab configuration for microtensile bond strength test.*

Table 1: *Means and Standard Deviation for Microtensile Bond Strength Values*

	n	Thermal Cycling (2,000 cycles 5°-55°C)	Mechanical Cycling (100,000 cycles - 50N)	Mean (SD)* MPa	p
G1	20	-	-	28.15 (14.03)a	0.825
G2	19	+	-	27.60 (10.14)a	0.994
G3	20	-	+	27.59 (8.67)a	0.823
G4	20	+	+	22.41 (8.57)b	0.024

(+ = performed; - = not performed)

**Different superscripted letters indicate statistically significant differences (p<0.05)*

RESULTS

Microtensile Bond Strengths

Statistically significant differences were observed between groups ($p<0.05$) as described in Table 1. Bond strengths were significantly lower when thermal and mechanical cycling were performed on the same specimens (G4- 22.4 ± 8.6 MPa) when compared to the other groups [Control (G1)- 28.2 ± 14.0 MPa; Thermal cycling (G2)-27.6 ± 10.1 MPa; Mechanical load cycling (G3)- 27.6 ± 8.7 MPa]. No differences were observed among the control, thermal cycling only and mechanical cycling only groups.

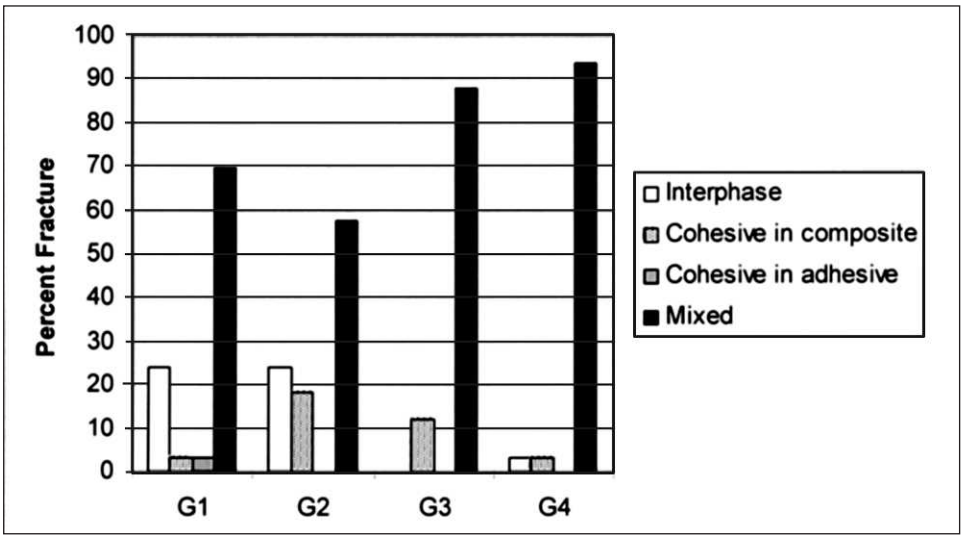


Figure 3. *Fracture mode analysis of the groups evaluated. G1- control, G2- Thermal cycling, G3- Mechanical load cycling, G4- Thermal and Mechanical load cycling.*

Fracture Mode Analysis

Figure 3 shows the fracture mode analysis results. The four types of failure modes [interphase, cohesive in composite, cohesive in adhesive and mixed] were observed. Failure at the interphase was observed mostly for the control group (G1) (Figure 4A and B) and the thermal cycling group (G2). Cohesive failure in composite was observed in all groups, especially G2 (Thermal cycling). Cohesive failure in the adhesive was only observed for the control group specimens at a very low percentage rate. Mixed failure was predominant and increased when mechanical cycling was applied (G3 and G4). The use of thermal cycling increased the cohesive failure of resin, reduced the mixed failures and presented similar interface failure when compared to the control group. When mechanical load cycling was performed alone, no interfacial failure was observed and mixed failure increased. The same behavior was observed when thermal and mechanical load cycling were performed on the same specimens (G4), resulting in more than 90% mixed failures.

Fracture at the interphase, showing the tags in parallel directions (Figure 5A), was also observed especially in the mechanical cycling groups (G3 and G4) and indicates a failure in the hybrid layer. Water voids within the interphase were observed in several specimens (Figure 5B) and could have some effect on bond strength values.

DISCUSSION

The goal in adhesive dentistry is to develop materials that are able to replace and act as biological hard dental structure. Sealing ability (nanoleakage and microleakage) and bond strength (tensile and shear bond strength) are the two main types of laboratory tests used to evaluate the performance of restorative systems. The feasibility of using these tests to evaluate restorations placed in cavity preparations permits the evaluation of a specific material in a condition that is clinically relevant. With the introduction of microtensile bond testing, it became possible to test clinically relevant substrates such as Class I, II and V preparations, caries-affected dentin and sclerotic dentin. The advantage of the microtensile bond test is to allow for the evaluation of small areas and different substrates (Sano & others, 1994; Pashley & others, 1999), which would not be possible using conventional shear and tensile tests.

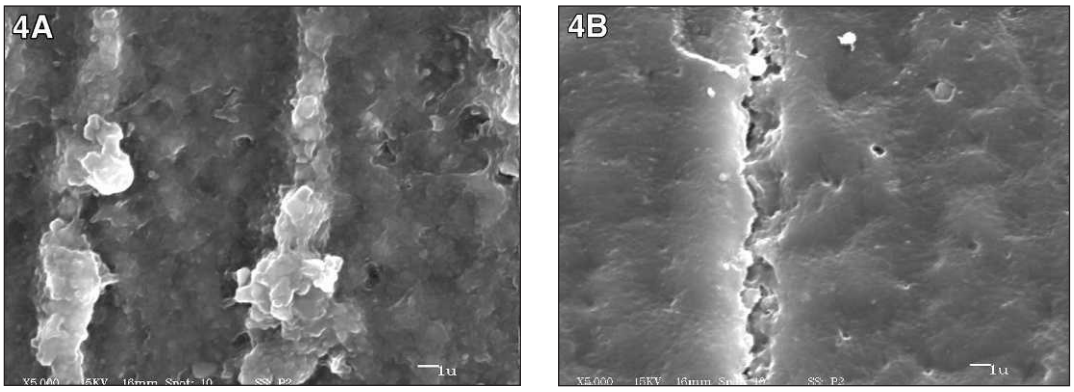


Figure 4. *A- High magnification of the interphase failure mode at dentin side. The parallel direction of the tubule and tags can be observed. B- High magnification of the interphase failure mode at restoration side. The parallel direction of the dentin tubules can be observed through grooves at the interface of fracture.*

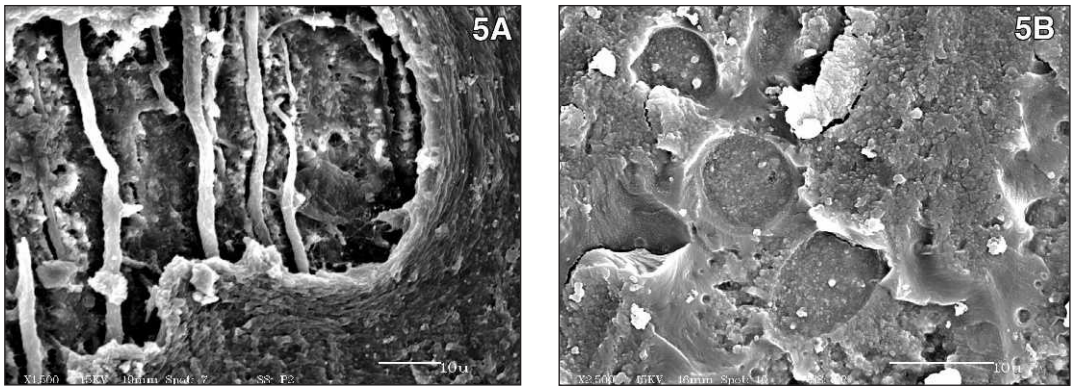


Figure 5. *A- Fracture at the interphase shows tags in parallel directions. B- SEM micrograph of voids within the interphase observed in several specimens.*

It has been reported that tensile bond strengths were lower at the apical wall compared to the occlusal walls of cervical wedge-shaped cavities (Ogata & others, 1999). Most of these differences can be explained by the direction of the tubules (Ogata & others, 2001). They demonstrate that for tubules oriented parallel to the bonded interface, higher values were observed when compared to tubules oriented perpendicular to the interface. A thicker hybrid layer was observed for the group with parallel dentin tubule orientation. In this study, dentin tubules were oriented parallel to the cervical wall, which allows for acceptable values of bond strength but which are still lower when compared to flat surface. One explanation may be the effect of the cavity configuration factor (C-factor) on bond strength data. C-factor is the ratio of the bonded surface area to the unbounded or free surface area (Feilzer, de Gee & Davidson, 1987). Bouillaguet and others (2001) reported a 20% reduction in bond strength of Class II cavity walls compared to flat dentin surfaces.

In the fracture mode analysis, the presence of water voids at the fracture interface was used (Figure 5B). During treatment of the cavity, due to its configuration, it may have been difficult to control the blot drying and application of the adhesive system. Zheng and others (2001) studied the influence of adhesive layer thickness. In their study, using an ethanol/water-based adhesive system (Single Bond) resulted in a decrease in bond strength values for thicker adhesive layers. According to the authors, in thicker layers, the solvent may not evaporate, leading to poor polymerization and a subsequent decrease in bond strength. Also, the use of a moist bonding technique at cavities makes blot drying a critical step, and the presence of excess water may hinder evaporation of the solvent. According to our data, it is difficult to know whether incomplete evaporation of the solvent, the presence of excess water in demineralized dentin or the association of both were responsible for the voids at the interface.

In this study, bovine incisors were used in place of human teeth. Previous studies (Nakamichi, Iwaku & Fusayama, 1983; Reeves & others, 1995) have shown the equivalent of bovine and human teeth in bonding tests. The major advantage of using bovine teeth is the ability to control age, sclerosis and the amount of wear of the substrate (average age of two years).

The use of thermocycling in *in vitro* mechanical test evaluation has been thoroughly investigated (Prati & others, 1994; Miyazaki & others, 1998; Hakimeh & others, 2000). The quantity of cycles and the temperatures used seem to be the major difference among different studies (Prati & others, 1994; Alani & Toh, 1997; Hakimeh & others, 2000). Controversial results have been reported regarding the influence of thermal cycling on microleakage. Darbyshire and others (1988); Prati and others (1994) and Chan and Glyn-Jones (1994) reported no effect of thermocycling on microleakage. Recently, Hakimeh and others (2000) reported increased microleakage in Class V restorations after 2880 cycles (4°C to 60°C).

Some studies report no influence of thermal stress when using a low number of cycles for shear bond strength (Miyazaki & others, 1998; Yoshida & Atsuta, 1999). Nikaido and others (2002b) observed no influence of 2,000 thermal cycles (5°C-55°C) on μTBS of flat dentin surface. The same authors observed a decrease in bond strength when thermocycling was performed in Class I preparations. However, these same authors used concomitant load cycling; therefore, it is difficult to evaluate the real influence of thermal cycling only on the outcome of the data. In this study, the influence of thermal cycling alone was not observed, but the use of thermal cycling and mechanical cycling in the same specimens reduced bond strength significantly.

The use of mechanical load cycling has been studied due to the potential capability of simulating mastication. In this study, a cylindrical polyacetal tip that touched only the material and aimed to fatigue the restoration, was used. A force of 50 N was chosen in order to simulate an average of constant load found during mastication (Anderson, 1956).

The evaluation of mechanical load cycling on μTBS from cavity pulp floors was recently documented (Nikaido & others, 2002a;b). Nikaido and others (2002a) evaluated the influence of 50,000 cycles on Class I preparations restored with self-etching or total etch adhesive systems. However, the authors did not report the influence of mechanical load cycling on μTBS. In another study, Nikaido and others (2002b), using the same load force (50N) and number of cycles (100,000) on Class I restorations employed in this study and associated with concomitant thermocycling, related a decrease in μTBS of a self-etching adhesive system. According to the current study, only the use of concomitant thermal cycling with mechanical load cycling could influence the results.

The use of different adhesive systems and cavity shapes may explain the different results found. Adhesive systems have been shown to behave differently according to the substrate to which they are bonded (Yoshikawa & others, 1999) and to operator variation (Finger & Balkenhol, 1999). The cavity shape used in this study permitted evaluation of an interface where dentin tubules were parallel. This may have been the reason for the higher bond strength values compared to tubules directed perpendicular (pulp floor) to the interface as reported by Nikaido and others (2002a). Also, in Class II cavities, the restoration is exposed to a lower C-factor than Class I restorations, which could be associated with the better bond strength values for Class II restorations.

According to the fracture mode analysis, the use of thermal cycling increased the cohesive failure of resin, reduced the mixed failures and presented similar interface failure when compared to the control group. The results of this study suggest that thermal cycling promoted a slight change in fracture mode but not enough to influence the μTBS values (Group 2). When mechanical load cycling was performed alone, no interfacial failure was observed and mixed failure increased. The same behavior was observed when thermal and mechanical load cycling were performed on the same specimens (Group 4), resulting in more than 90% mixed failures. It can be speculated that for this group, because thermal cycling was performed first, it promoted stress on the interface. Then, mechanical cycling was applied to these specimens, which had already been exposed to a modified environment due to thermocycling. Consequently, the effect of loading was accelerated by the thermocycling, resulting in significantly lower bond strength. The mixed fracture mode consisted of three types of fracture, whether or not they appeared together. It is difficult to specify the localized stress point at the interface. Observing that fracture at the interphase was reduced with load cycling, it can be suggested that mechanical loading could weaken the adhesive resin at the bonded interface.

Weakening the adhesive resin due to mechanical loading is a very important issue in restorative dentistry since the demand for posterior composites by patients has significantly increased. The recent adhesive technologies that combine primer and adhesive may be at greater risk for failure if the solvent is not adequately removed by air drying. Although technique sensitivity and type of adhesive resin may vary, future studies should focus on determining the durability of adhesive resins under mechanical loading.

CONCLUSIONS

The null hypothesis that thermal cycling and mechanical load cycling would not influence bond strength was rejected when both were used in the same specimens, reducing bond strength.

Thermal and mechanical cycling combined adversely affected bond strengths and failure modes. Simulation of the oral condition might be crucial to better evaluate and understand the performance of adhesive materials.

Acknowledgements

This study was supported in part by Research Grant #01515/01-2 from Capes/Brazil.

(Received 3 February 2003)

References

Abdalla AI & Davidson CL (1993) Comparison of the marginal integrity of *in vivo* and *in vitro* Class II composite restorations *Journal of Dentistry* **21(3)** 158-162.

Abdalla AI & Davidson CL (1996) Effect of mechanical load cycling on the marginal integrity of adhesive Class I resin composite restorations *Journal of Dentistry* **24(1-2)** 87-90.

Alani AH & Toh CG (1997) Detection of microleakage around dental restorations: A review *Operative Dentistry* **22(4)** 173-185.

Anderson DJ (1956) Measurement of stress in mastication I *Journal of Dental Research* **35(5)** 664-670.

Ausiello P, Davidson CL, Cascone P, de Gee AJ & Rengo S (1999) Debonding of adhesively restored deep Class II MOD restorations after functional loading *American Journal of Dentistry* **12(2)** 84-88.

Bouillaguet S, Ciucchi B, Jacoby T, Wataha JC & Pashley D (2001) Bonding characteristics to dentin walls of Class II cavities, *in vitro Dental Materials* **17(4)** 316-321.

Chan MF & Glyn-Jones JC (1994) Significance of thermal cycling in microleakage analysis of root restorations *Journal of Dentistry* **22(5)** 292-295.

da cunha Mellow FS, Feilzer AJ, de Gee AJ & Davidson CL (1997) Sealing ability of eight resin bonding systems in a Class II restoration after mechanical fatiguing *Dental Materials* **13** 372-376.

Darbyshire PA, Messer LB & Douglas WH (1988) Microleakage in Class II composite restorations bonded to dentin using thermal and load cycling *Journal of Dental Research* **67(3)** 585-587.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Finger WJ & Balkenhol M (1999) Practitioner variability effects on dentin bonding with an acetone-based one-bottle adhesive *Journal of Adhesive Dentistry* **1(4)** 311-314.

Hakimeh S, Vaidyanathan J, Houpt ML, Vaidyanathan TK & Von Hagen S (2000) Microleakage of compomer Class V restorations: Effect of load cycling, thermal cycling, and cavity shape differences *Journal of Prosthetic Dentistry* **83(2)** 194-203.

Jorgensen KD, Itoh K, Munksgaard EC & Asmussen E (1985) Composite wall-to-wall polymerization contraction in dentin cavities treated with various bonding agents *Scandinavian Journal of Dental Research* **93(3)** 276-279.

Miyazaki M, Sato M, Onose H & Moore BK (1998) Influence of thermal cycling on dentin bond strength of two-step bonding systems *American Journal of Dentistry* **11(3)** 118-122.

Nakabayashi N & Pashley DH (1998) Evolution of dentin resin bonding in *Hybridization of Dental Hard Tissues* Quintessence Publishing Co Ltd Tokyo 1-17.

Nakamichi I, Iwaku M & Fusayama T (1983) Bovine teeth as possible substitutes in the adhesion test *Journal of Dental Research* **62(10)** 1076-1081.

Nara Y, Suzuki T, Kizuki I, Miyamoto M, Kimishima T, Maseki T, Tanaka H & Dogon L (2002) Effect of thermal cycling and/or repeated load on microleakage of cervical composite restoration *Journal of Dental Research* **81** A-418 (#3387).

Nikaido T, Kunzelmann KH, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R & Tagami J (2002a) The *in vitro* bond strengths of two adhesive systems in Class I cavities of human molars *Journal of Adhesive Dentistry* **4(1)** 31-39.

Nikaido T, Kunzelmann KH, Chen H, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R & Tagami J (2002b) Evaluation of thermal cycling and mechanical loading on bond strength of a self-etching primer system to dentin *Dental Materials* **18(3)** 269-275.

Ogata M, Nakajima M, Sano H & Tagami J (1999) Effect of dentin primer application on regional bond strength to cervical wedge-shaped cavity walls *Operative Dentistry* **24(2)** 81-88.

Ogata M, Okuda M, Nakajima M, Pereira PN, Sano H & Tagami J (2001) Influence of the direction of tubules on bond strength to dentin *Operative Dentistry* **26(1)** 27-35.

Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1(4)** 299-309.

Prati C, Tao L, Simpson M & Pashley DH (1994) Permeability and microleakage of Class II resin composite restorations *Journal of Dentistry* **22(1)** 49-56.

Reeves GW, Fitchie JG, Hembree JH Jr & Puckett AD (1995) Microleakage of new dentin bonding systems using human and bovine teeth *Operative Dentistry* **20(6)** 230-235.

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.

Yoshida K & Atsuta M (1999) Effect of MMA_PMMA resin polymerization initiators on the bond strengths of adhesive primers for noble metal *Dental Materials* **15(5)** 332-336.

Yoshikawa T, Sano H, Burrow MF, Tagami J & Pashley DH (1999) Effects of dentin depth and cavity configuration on bond strength *Journal of Dental Research* **78(4)** 898-905.

Zheng L, Pereira PN, Nakajima M, Sano H & Tagami J (2001) Relationship between adhesive thickness and microtensile bond strength *Operative Dentistry* **26(1)** 97-104.

©Operative Dentistry, 2004, **29-2,** 157-161

Effect of Light Curing Method on Volumetric Polymerization Shrinkage of Resin Composites

MJM Coelho Santos • GC Santos, Jr • H Nagem Filho
RFL Mondelli • O El-Mowafy

Clinical Relevance

Following completion of exposure, the volumetric polymerization shrinkage of three different light-activated resin composites were not affected when various light exposure modes were applied: continuous output using conventional intensity, continuous output using a slightly higher intensity and two soft-start methods—a ramped output and a pulse-delay method.

SUMMARY

Volumetric polymerization shrinkage of three resin composites (Suprafil, Z100 and Filtek P60) was determined using four light curing methods: method 1: continuous output with conventional intensity light; method 2: continuous output with higher intensity light; method 3: ramp output and method 4: pulse-delay output. Five disc-shaped specimens were prepared from each material for each curing method. Specimen weight was determined with an analytical electronic hydrostatic balance in air and in water before and after curing. Specific gravity values were then determined. Volumetric polymerization shrinkage was calculated using mathematical formulas. Mean volumetric polymerization shrinkage ranged from 1.882 (.015)% to 2.169 (.028)%. ANOVA indicated significant differences among the materials ($p<.05$). Light curing methods had no effect on volumetric polymerization shrinkage except for Z-100, where method 2 resulted in significantly higher shrinkage than methods 1 and 4. Suprafil shrunk significantly less than the other two materials in all curing methods.

Maria Jacinta Moraes Coelho Santos, DDS, MS, PhD, associate professor, Department of Operative Dentistry, University of Bahia, School of Dentistry, Salvador, BA, Brazil

Gildo Coelho Santos, Jr, DDS, MS, PhD, associate professor, Department of Operative Dentistry, University of Bahia, School of Dentistry, Salvador, BA, Brazil

Halim Nagem Filho, DDS, MS, PhD, associate professor, Department of Operative Dentistry, Bauru School of Dentistry, University of Sagrado Coração, SP, Brazil

Rafael Francisco Lia Mondelli, DDS, MS, PhD, associate professor, Department of Operative Dentistry, Bauru School of Dentistry, University of São Paulo, SP, Brazil

*Omar El-Mowafy, BDS, PhD, FADM, associate professor, Discipline of Restorative Dentistry, Department of Clinical Sciences, Faculty of Dentistry, University of Toronto, Toronto, Ontario, Canada

*Reprint request: 124 Edward Street, Toronto, Ontario M5G 1G6, Canada; e-mail: oel.mowafy@ utoronto.ca

INTRODUCTION

Polymerization shrinkage is an inherent property of current resin composites, whether they are self- or light-cured. Monomer conversion into polymer results in a closer, tighter arrangement of molecules leading to a reduction in material volume. Intermolecular distance changes from 0.3-0.4 nm to 0.15 nm on polymerization of resin composite (Peutzfeldt, 1997).

Volumetric polymerization shrinkage values have been reported to range from 0.9% to 2.24% for six resin composites (Rees & Jacobson, 1989), from 1.35% to 3.22% for six light-cured posterior resin composites (Puckett & Smith, 1992) and from 2.1% to 2.7% for three polyacid-modified resin composites (Miyazaki, Fukuishi & Onose, 1999). A number of factors can influence polymerization shrinkage. These include inorganic filler content, (Munksgaard, Hansen & Kato, 1987; Iga & others, 1991) (the more the inorganic filler content, the less the shrinkage), type of monomer and degree of cure and light intensity/curing cycle (Miyazaki & others, 1999; Unterbrink & Muessner, 1995; Feilzer & others, 1995; Dennison & others, 2000; Sakaguchi, Douglas & Peters, 1992).

Tarle and others (1998) showed that use of a pulsed laser light for 40 seconds reduced polymerization shrinkage of two composite materials, while at the same time resulting in a higher degree of monomer conversion. However, reduction in polymerization shrinkage occurred only in comparison with 120 seconds of conventional light curing but not with 40 seconds, which is the duration typically recommended by manufacturers of composite materials.

Polymerization shrinkage can be reduced through limiting the degree of monomer conversion; however, this reduction will have adverse effects on the physical and mechanical properties of restorations. Maximum monomer conversion is always desired to ensure optimum properties and biocompatibility and reduce water solubility (Asmussen, 1982; Ferracane & Greener, 1986; Venhoven, de Gee & Davidson, 1993; Davidson-Kaban & others, 1997).

A curing cycle that uses initial low-intensity irradiance followed by final higher intensity value was found to achieve better marginal adaptation with resin composite restorations (Mehl, Hickel & Kunzelmann, 1997; Koran & Kurschner, 1998; Kanca & Suh, 1999; Kinomoto & others, 1999). Among the methods used to achieve this reduction were ramp-cure (soft-start) and pulse-cure. The two methods have in common an initial, short, low-intensity light application followed by final exposure at a higher intensity. In a recent study, different pulse activation and soft-start cycles were used to test their effect on the hardening of a resin composite (Yap, Soh & Siow, 2002). Some pulse delay methods resulted in inferior hardness values compared to a control group that used continuous output. In contrast, Dennison and others (2000) found that some alternative exposure techniques can be useful in reducing linear polymerization shrinkage of resin composites without compromising depth of cure. However, their work dealt with a conventional intensity light unit (Optilux model VCL 401, Demetron Research Corp, Danbury, CN, USA) and low light intensity irradiance cycles (25% and 50% of the full intensity of the light). It is important to determine the effect of higher light intensity levels on polymerization shrinkage when alternative curing methods are used.

This investigation determined the effect of different light intensities and curing scenarios on volumetric polymerization shrinkage of a variety of commercially available resin composites.

METHODS AND MATERIALS

The three hybrid resin composites included in this study are described in Table 1. A stainless steel ring 6-mm in diameter and 2-mm thick was used, together with a glass section and Mylar strip, to shape specimens made from these materials into discs. Five specimens were prepared from shade A2 of each material for each of four curing methods. Each specimen was expressed carefully from one side of the mold and weighed twice in an analytical electronic hydrostatic balance with an accuracy of 0.0001 g and a specific gravity measuring kit (Analytical balance AND, model AD-1653, A & D Company Ltd, Tokyo, Japan). The specimens were first weighed in air, then in distilled water following a method similar to that used by Puckett and Smith (1992). Water temperature was maintained at 23°C. Water temperature was measured prior to each testing using a digital thermometer (HI 93530, Gehaka, Brazil). Specimens were dried carefully with absorbent paper and then subjected to one of the following four light curing methods: method 1: continuous output with conventional intensity light at 500mW/cm^2 for 60 seconds; method 2: continuous output with higher intensity light at 700mW/cm^2 for 60 seconds; method 3: ramp output from 100 to 700 mW/cm^2 in 15 seconds, then 45 seconds at 700mW/cm^2; method 4: pulse-delay output 3 seconds at 200 mW/cm^2 followed by three minutes wait, then 30 seconds at 600 mW/cm^2. Two light curing units were used for this purpose (methods 1 & 3: Elipar TriLight, ESPE, Norristown, PA, USA; and methods 2 and 4: BISCO VIP, BISCO Dental Products, Schaumburg, IL, USA). Specimens were weighed immediately after light curing. The weight values for each specimen in air and water were used to calculate specific gravity values before and after curing. These values were used to determine volumetric shrinkage according to the following formulas (Puckett & Smith, 1992):

SG 1 = M1/(M1-M2) (1); SG2 = M3/(M3-M4) (2)

SG 1 = specific gravity before polymerization

SG 2 = specific gravity after polymerization

M1 = mass of specimen weighed in air before polymerization

M2 = mass of specimen weighed in the water before polymerization

M3 = mass of specimen weighed in air after polymerization

M4 = mass of specimen weighed in the water after polymerization

Density of specimen (kg/cm3) = SG x density of the water at 23°C

V1 = M1/D1 (3) V2 = M3/D2 (4)

V1 = Volume of specimen before polymerization

V2 = Volume of specimen after polymerization

D1 = Density of specimen before polymerization

D2 = Density of specimen after polymerization

Percentage contraction = (V2 - V1)/V2 x 100 (5)

Means and standard deviations for volumetric polymerization shrinkage were determined for each group and data were statistically analyzed using a two-way ANOVA followed by the Tukey's test at 5% level of significance.

RESULTS

Mean and standard deviation values for volumetric polymerization shrinkage of the three resin composites with the four curing methods are shown in Table 2. ANOVA revealed significant differences in mean polymerization shrinkage values among the groups ($p<.05$). Significant differences were detected among materials (Table 2) and curing methods. Tukey's test showed that a significantly higher polymerization shrinkage value [2.169 (.028)] was found only with Z-100 when a higher intensity light curing was used (method 2). However, this increase in shrinkage was only 5% higher than the value obtained with conventional intensity curing [2.065 (-029)]. When ramp curing was used with Z-100, shrinkage was 2.104 (.028). This value was not significantly different from values obtained with this material when the other two exposure methods were used (methods 1 and 4). Compared to the other two materials, Suprafil had lower shrinkage using all four curing methods. However, shrinkage of this material was only 6% to 9% lower than the values recorded for Filtek P-60 and 8% to 13% lower than the values recorded for Z-100. Shrinkage values of Filtek P 60 and Suprafil using the four curing methods were not significantly different.

DISCUSSION

Many methods have been used to measure volumetric polymerization shrinkage of resin composites, with most reports using dilatometers (with mercury or water). The major problem with this method is the difficulty in getting the light source to reach the immersed composite specimen and, in the case of mercury dilatometer, the manipulation of hazardous material (Watts & Cash, 1991). The density measurement for determining volumetric shrinkage is a simple, accurate and reproducible procedure that does not require the use of elaborate equipment.

The volumetric polymerization shrinkage values presented in this study are similar to those reported by Puckett and Smith (1992) who, using the same measurement technique, examined a group of six different resin composites and reported values ranging from 1.35% to 3.22%. However, Dennison and others (2000), who determined the linear polymerization shrinkage of two resin composites with different curing methods, reported a range of .56% to 1.50%. They used a mathematical formula to calculate volumetric shrinkage from linear shrinkage and reported a range from 1.67% to

Table 1: *Resin Composite Materials Used (Composition Information Provided by Manufacturers)*

Material	Lot #	Manufacturer	Resin Matrix	Fillers	Filler Content (Weight)
Z-100	2BR	3M Dental Products, St Paul, MN, USA	Bis-GMA TEGDMA	Zirconia Silica	84.5%
Filtek P-60	2WE	3M Dental Products, St Paul, MN, USA	Bis-GMA Bis-EMA UDMA	Zirconia Silica	83.0%
Suprafill	00R	SS-White, Gloucester, GL1-5SG, England	Bis-GMA TEGDMA UDMA	Ba_2SiO_4	76.5%

Table 2: *Mean Volumetric Polymerization Shrinkage (SD) for the Three Composite Materials Using the Four Curing Methods. Means Within the Vertical Lines Are Not Significantly Different*

	Photocuring Method			
Material	**1. Higher Intensity**	**2. Conventional**	**3. Ramp Cycle**	**4. Pulse Cycle**
Filtek P 60	2.006 (.038)	2.058 (.021)	2.050 (.025)	2.020 (.019)
Z-100	2.169 (.028)	2.065 (.029)	2.104 (.028)	2.071 (.021)
Suprafill	1.882 (.015)	1.88 (.006)	1.886 (.001)	1.836 (.001)

4.42% for two materials. This range is slightly wider than the current study and one by Puckett and Smith (1992). It is likely that this discrepancy might be related to the fact that different materials were studied in each of the three studies.

Using the density method, Unterbrink and Muessner (1995) reported volumetric shrinkage of two resin composites (Tetric and Z100) using different light intensities (450 mW/cm^2 and 250 mW/cm^2). For Z-100, they reported mean shrinkage values of 2.41% and 2.36% with higher and lower light intensity, respectively, when the test was conducted five minutes after polymerization. These values compare well with those for the same material reported in this study: 2.17% and 2.07% with higher (700 mW/cm^2) and conventional (500 mW/cm^2) intensity, respectively. The small variance obtained in the present study and, indeed, with other materials in studies by Unterbrink and Muessner (1995) and Puckett and Smith (1992) indicate that the density method is accurate and reproducible.

Miyazaki and others (1999), who used a dilatometer method, reported a positive correlation between the volumetric polymerization shrinkage of three compomers and light intensity. However, an increase in intensity from 300 to 600 mW/cm^2 resulted in a significant increase in polymerization shrinkage with some, but not all materials. This finding compares well with those of the current study with two materials showing no significant difference in shrinkage when light intensity was increased from 500 to 700 mW/cm^2, while the third material showed a significant difference.

The four methods of light curing of this study resulted in four different values of total energy. Methods 1 and 2 resulted in total energy values of 30,000 mJ/cm^2 and 42,000 mJ/cm^2, respectively, while with the ramped output, an approximate total energy of 36,500 mJ/cm^2 resulted. Method 4 resulted in a total energy value of only 18,600 mJ/cm^2. According to Rueggeberg, Caughman and Curtis (1994), a light intensity of 400 mW/cm^2 for 40 seconds would be sufficient to cure a resin composite increment 2-mm thick, representing a total energy of 16,000 mJ/cm^2. Therefore, all four curing methods used here resulted in total energy values greater than what one would use clinically. However, in spite of the variability in total energy of the four curing methods, this difference had little or no effect on volumetric polymerization shrinkage for most resin composite/curing method combinations.

In spite of a lower inorganic filler content by Suprafil (76.5% by weight compared with 84.5% for Z 100 and 83% for P-60), it demonstrated significantly lower shrinkage than the other two materials. This result could be related to a higher amount of Bis-GMA content in this material relative to the other monomers used (TEGDMA and UEDMA). Bis-GMA undergoes lower polymerization shrinkage due to its high molecular weight and low degree of conversion (Peutzfeldt, 1997). Incorporation of di-methacrylate diluents by the manufacturer is necessary to reduce the high viscosity of the Bis-GMA and allows for the incorporation of more inorganic fillers (Peutzfeldt, 1997). These diluent monomers have low molecular weight and are more reactive and, as a result, are the major cause of polymerization shrinkage (Peutzfeldt, 1997). UEDMA has similar molecular weight as Bis-GMA (Peutzfeldt, 1997), and its presence in the composition of Suprafil might have helped to reduce polymerization shrinkage.

Koran and Kurschner (1998) evaluated the effect of continuous and step light curing methods on polymerization shrinkage of resin composites. They reported that, although the dynamics of polymerization shrinkage was better with the step light curing technique, the overall final polymerization shrinkage was equal for the two curing methods.

The slower development of polymerization shrinkage with the use of gradual curing modes, ramp or pulse output, results in better marginal adaptation of resin composite restorations (Mehl and others, 1997; Kanca & Suh, 1999; Kinomoto & others, 1999). Perhaps these soft start methods allow for better arrangement of molecules at the marginal areas during the pre-gel phase, as it has been suggested that stress build up at the tooth/restoration interface, rather than actual polymerization shrinkage, is the main cause for marginal adhesive failure (Davidson & Feilzer, 1997; Feilzer, de Gee & Davidson, 1987). However, Yap and others (2002), who investigated the effectiveness of composite cure with pulse-delay and soft-start polymerization techniques, concluded that using some of these alternative soft-start techniques may result in reduced hardness of the bottom surface of a 2-mm increment of composite compared to conventional intensity and continuous exposure. Further research in this area is necessary in order to reveal the best curing cycle that would result in optimum polymerization of a resin composite increment with the least possible amount of shrinkage.

CONCLUSIONS

1. The light curing methods used in this study had no significant effect on the volumetric polymerization shrinkage of three resin composites except for Z-100. However, the increase in shrinkage of Z-100 when higher intensity light was used continuously was small compared with shrinkage obtained with conventional light intensity.
2. Suprafil showed lower polymerization shrinkage values than the other two materials with all curing methods.

(Received 3 February 2003)

References

Asmussen E (1982) Restorative resins: Hardness and strength vs quantity of remaining double bonds *Scandinavian Journal of Dental Research* **90(6)** 484-496.

Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restoratives *Journal of Dentistry* **25(6)** 435-440.

Davidson-Kaban SS, Davidson CL, Feilzer AJ, de Gee AJ & Erdilek N (1997) The effect of curing light variations on bulk curing and wall-to-wall quality of two types and various shades of resin composites *Dental Materials* **13(6)** 344-352.

Dennison JB, Yaman P, Seir R & Hamilton JC (2000) Effect of variable light intensity on composite shrinkage *The Journal of Prosthetic Dentistry* **84(5)** 499-505.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Feilzer AJ, Dooren LH, de Gee AJ & Davidson CL (1995) Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface *European Journal of Oral Sciences* **103(5)** 322-326.

Ferracane JL & Greener EH (1986) The effect of resin formulation on the degree of conversion and mechanical properties of dental restorative resins *Journal of Biomedical Materials Research* **20(1)** 121-131.

Iga M, Takeshige F, Ui T & Torii M (1991) The relationship between polymerization shrinkage measured by a modified dilatometer and the inorganic filler content in light cured composites *Dental Materials Journal* **10(1)** 38-45.

Kanca J 3rd, & Suh BI (1999) Pulse activation: Reducing resin-based composite contraction stresses at the enamel cavosurface margins *American Journal of Dentistry* **12(3)** 107-112.

Kinomoto Y, Torii M, Takeshige F & Ebisu S (1999) Comparison of polymerization contraction stresses between self and light-curing composites *Journal of Dentistry* **27(5)** 383-389.

Koran P & Kurschner R (1998) Effect of sequential versus continuous irradiation of a light-cured resin composite on shrinkage, viscosity, adhesion, and degree of polymerization *American Journal of Dentistry* **11(1)** 17-22.

Mehl A, Hickel R & Kunzelmann KH (1997) Physical properties and gap formation of light-cured composites with and without "softstart-polymerization" *Journal of Dentistry* **25(3-4)** 321-330.

Miyazaki M, Fukuishi K & Onose H (1999) Influence of light irradiation on the volumetric change of polyacid-modified resin composites *Journal of Dentistry* **27(2)** 149-152.

Munksgaard EC, Hansen EK & Kato H (1987) Wall-to-wall polymerization contraction of composite resin versus filler content *Scandinavian Journal of Dental Research* **95(6)** 526-531.

Peutzfeldt A (1997) Resin composites in dentistry: The monomer systems *European Journal of Oral Sciences* **105(2)** 97-116.

Puckett AD & Smith R (1992) Method to measure the polymerization shrinkage of light-cured composites *The Journal of Prosthetic Dentistry* **68(1)** 56-58.

Rees JS & Jacobson PH (1989) The polymerization shrinkage of composite resins *Dental Materials* **5(1)** 41-44.

Sakaguchi RL, Douglas WH & Peters MC (1992) Curing light performance and polymerization of composite restorative materials *Journal of Dentistry* **20(3)** 183-188.

Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.

Tarle Z, Meniga A, Ristic M, Sutalo J, Pichler G & Davidson CL (1998) The effect of the photopolymerization method on the quality of composite resin samples *Journal of Oral Rehabilitation* **25(6)** 436-442.

Unterbrink GL & Muessner R (1995) Influence of light intensity on two restorative systems *Journal of Dentistry* **23(3)** 183-189.

Venhoven BA, de Gee AJ & Davidson CL (1993) Polymerization contraction and conversion of light-curing Bis-GMA based methacrylate resins *Biomaterials* **14(11)** 871-875.

Watts DC & Cash AJ (1991) Determination of polymerization shrinkage kinetics in visible-light-cured materials: Methods development *Dental Materials* **7(4)** 281-287.

Yap AUJ, Soh MS & Siow KS (2002) Effectiveness of composite cure with pulse activation and soft-start polymerization *Operative Dentistry* **27(1)** 44-49.

©Operative Dentistry, 2004, **29-2,** 162-167

The Effect of Flowable Resin Composites as Gingival Increments on the Microleakage of Posterior Resin Composites

N Attar • MD Turgut • HC Güngör

Clinical Relevance

The use of a flowable composite or compomer as the first gingival increment of Class II restorations either with microhybrid or packable composites decreased gingival microleakage regardless of the material used.

SUMMARY

Microleakage has been a major concern in restorative dentistry. The curing contraction of composites still presents a problem with controlling microleakage and postoperative sensitivity. This study investigated the effect of flowable materials on gingival microleakage of microhybrid and packable resin composite restorations.

Ninety Class II cavities with cervical margins 1 mm below the CEJ were prepared in 45 extracted human premolars. The teeth were randomly divided into three groups (n=15). In each group, one side of each tooth was restored incrementally with respective composites–SureFil, Filtek P60 and Tetric Ceram; whereas, on the other side, flowable materials–Dyract Flow, Filtek Flow or Tetric Flow–were placed respectively as a 1-mm thick gingival increment before resin composite restoration. The restored teeth were stored for one week in distilled water at 37°C, thermocycled between 5°C and 55°C and immersed in 0.5% basic fuchsin for 24 hours. Dye penetration was evaluated using a stereomicroscope at 10x magnification. The data were analyzed statistically by Kruskal-Wallis analysis of variance and Mann-Whitney U-tests. The effect of flowable increments on reducing the gingival microleakage was found to be statistically significant for all restorative materials tested ($p<0.05$).

*Nuray Attar, DDS, PhD, research assistant, Department of Conservative Dentistry, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

Melek D Turgut, DDS, PhD, research assistant, Department of Pedodontics, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

H Cem Güngör, DDS, PhD, research assistant, Department of Pedodontics, Faculty of Dentistry, Hacettepe University, Ankara, Turkey

*Reprint request: Emek 8 Cadde, Buket A Apartmanı, No: 62 A Daire No: 12, 06510, Emek, Ankara, Turkey; e-mail: nurayattar@hotmail.com

INTRODUCTION

For the last two decades composite restorations have become a popular alternative to amalgam restorations in posterior teeth. The patients' demand for better esthetics, concerns related to possible mercury toxicity from amalgam and improvements in composite materials have significantly contributed the popularity of these materials (Leinfelder, 1991; Jackson & Morgan,

2000). Early problems related to composites included excessive wear, loss of anatomic form, post-operative sensitivity, secondary caries and marginal leakage. This has resulted, initially, in a diminished acceptance of these materials as suitable alternatives for the restoration of molars and premolars (Phillips & others, 1973; Leinfelder & others, 1980; Jackson & Morgan, 2000). The disadvantages of light cured composite materials with respect to microleakage are predominantly a result of polymerization shrinkage on curing. Depending on the amount of composite cured and the direction of the light source, polymerization shrinkage of composite materials may lead to gaps between the cavity walls and restoration. It has commonly been assumed that resins shrink toward the light and tend to polymerize away from the margins of the cavity, however, recent investigators have disputed this (Unterbrink & Muessner, 1995; Verluis, Tantbirojn & Douglas, 1998). It has been proposed that resin materials shrink toward the fixed boundaries and no significant differences in vector patterns were demonstrated between autocured and light cured composites (Feilzer, de Gee & Davidson, 1987).

Packable composites have been introduced to the dental market to overcome some of the problems associated with current composites. The increased viscosity of these materials permits for greater "packability" and these materials demonstrate less slumping characteristics. The manufacturers claim that these new materials demonstrate lower polymerization shrinkage as compared to conventional universal composites (Dentsply DeTrey, 1998; 3M Dental Products, 1999). They are characterized by a high–filler load and a filler distribution that gives them a different consistency compared to hybrid composites (Dentsply DeTrey, 1998; 3M Dental Products, 1999). Packable composites are indicated for stress bearing posterior restorations with improved handling properties, with an application technique similar to amalgam. Adaptation of the material to the tooth surface is dependent upon the viscosity of the restorative material and the adhesive technique used (Opdam & others, 1996). It was suggested that using flowable composites in combination with packable composites may help to demonstrate better marginal integrity (Bayne & others, 1998; Unterbrink & Liebenberg, 1999; Leevailoj & others, 2001; Beznos, 2001; Tung, Estafan & Scherer, 2000).

Flowable composites were created by retaining the same small particle sizes of traditional hybrid composites but reducing the filler content and allowing the increased resin to reduce the viscosity of the mixture (Bayne & others, 1998). They have been suggested for Class I, II, III and V cavity restorations, preventive resin restorations and composite, porcelain and amalgam repairing. Flowable composites can be used as liners, fissure sealants and also in tunnel preparations (Bayne & others, 1998; Estafan, Schulman & Calamia, 1999). Flowables create an intimate union with the microstructural defects in the floor and walls of the cavity preparation (Payne, 1999). They can be used to fill irregular internal surfaces and access difficult areas, such as in the gingival seat of an internal proximal box, especially when high viscosity composites are used. Their usage as a liner under high filled resins in posterior restorations has been shown to improve the adaptation of composites and effectively achieve clinically acceptable results (Unterbrink & Liebenberg, 1999).

This study investigated the effect of flowable materials on the gingival marginal leakage of two packable and one microhybrid resin.

METHODS AND MATERIALS

In this study, 45 non-carious human premolars were used. They were recently extracted and stored in saline at room temperature. A hand-scaling instrument was used for surface debridement of the teeth, which was followed by cleaning with a rubber cup and slurry of pumice. Standard Class II cavity preparations were placed by one operator on the mesial and distal surfaces of each tooth using a high-speed handpiece with air-water spray and a #1090 diamond fissure bur (Diatech Dental AG, Heerbrugg, Switzerland). New burs were used after every four preparations. The occlusal parts of the preparations were measured 1.5 mm in depth and 1.5 mm in buccolingual width. The proximal box margins of all cavities were placed 1 mm below the cementoenamel junction (CEJ). The depth of the box from cavosurface margin to the axial wall was 2 mm and the buccolingual width was 3.0 mm. The buccal and lingual walls

Table 1: *Resin Composites and Enamel-dentin Bonding Systems Used in the Study*

Group	Product		Batch #	Manufacturer
1	Bonding agent Flowable compomer Packable composite	Prime&Bond NT Dyract Flow SureFil	60667240 60604430 60605750	Dentsply, DeTrey GmbHD 78467 Konstanz Germany
2	Bonding agent Flowable composite Packable composite	Single Bond Filtek Flow Filtek P60	3411MP 8300A3 4720B2	3M Dental Products St Paul, MN, USA
3	Bonding agent Flowable composite Microhybrid composite	Excite Tetric Flow Tetric Ceram	B16833 546323AN 546307AN	Vivadent Ivoclar AG, FL- 9494, Schaan Liechtenstein

of the preparations were approximately parallel and connected to the gingival wall with rounded line angles. Cavosurface margins were prepared sharp without bevel. The root apices of the samples were sealed with a light cured hybrid resin composite (TPH, Dentsply-DeTrey, Konstanz, Germany) in order to reduce apical leakage into the pulp, which could confound the results.

The teeth were then randomly divided into three groups of 15 teeth each and restored with the tested materials according to the manufacturers' instructions. The materials used in the study are listed in Table 1. All preparations in each group were etched with each manufacturer's acid etching gel for 15 seconds, rinsed with water for 20 seconds and gently air dried to leave the surfaces wet (Ferrari & others, 1998).

Group 1: In this group, Prime & Bond NT (Dentsply-DeTrey) was applied to the wet surfaces with a brush, left undisturbed for 20 seconds and the excess solvent removed with a gentle stream of air. Light curing was done for 20 seconds with a visible light unit whose light intensity was 500 mW/cm^2 (Hilux 200 Curing Light, Benlioglu Dental Inc, Ankara, Turkey). One side of each tooth was restored incrementally with SureFil (Dentsply-DeTrey) (Group 1a) and with flowable compomer on the respective side; Dyract Flow (Dentsply-DeTrey) was carefully placed as a gingival increment in 1-mm thick. The remainder of this part was then restored with SureFil (Group 1b).

Group 2: The same procedures were followed as in the first group. Two coats of the bonding agent, Single Bond (3M Dental Products, St Paul, MN, USA), was applied to the wet surfaces with a brush; the excess solvent was removed with a gentle stream of air and light cured for 20 seconds. One side of each tooth was restored with packable composite Filtek P60 (3M Dental Products) (Group 2a); whereas, on the other side, gingival increment was placed with flowable composite Filtek Flow (3M Dental Products) and restored with Filtek P60 (Group 2b).

Group 3: In this group, the bonding agent, Excite (Vivadent Ets, Schaan, Liechtenstein), was applied to the preparations with a brush for 10 seconds, dried with a gentle stream of air and light cured for 20 seconds. The cavities were restored either with microhybrid composite Tetric Ceram (Vivadent Ets) (Group 3a) or Tetric Flow (Vivadent Ets) as in the other groups (Group 3b).

In each group, flowable materials were placed without matrix bands in order to easily manipulate and standardize the thickness of the increment. After light curing the increment for 40 seconds, a Tofflemire matrix retainer (Digument, Germany) and a metal band were placed on the tooth and tightly held by two fingers from the mesial and distal gingival margins. Composites were incrementally placed with a thickness of approximately 2 mm and each increment was light cured for 40 seconds. There was a minimal need for finishing, which was carried out using 30 blade carbide burs. Polishing of the samples was conducted with a series of Sof-Lex disks (3M Dental Products). Gingival margins were not disked. All samples were stored in distilled water at 37°C for one week, then thermocycled for 500 cycles between 5°C and 55°C with a dwell time of 30 seconds.

The teeth were painted with two coats of nail varnish 1 mm short of the margins to be exposed to dye. They were then immersed in 0.5% basic fuchsin dye for 24 hours at 37°C. After removal from the dye, the samples were cleaned under running tap water. The samples were sectioned mesiodistally through the center of the restorations with double-face diamond discs to obtain two sections from each tooth (Diamond Disc Superflex, 910S/220, North Bel, Italy). The sections were randomly arranged and assigned code numbers to permit blind evaluation. One operator evaluated the sections under a stereomicroscope (Wild Typ 308700, Heerbrugg, Switzerland) at 10x magnification. The following scoring scale (Leevailoj & others, 2001) was used to assess the extent of the dye penetration at the tooth-restoration interface (Figure 1): 0=no dye penetration, 1=dye penetration less than 1/3 of the gingival or occlusal wall, 2=dye penetration beyond 1/3 of the gingival or occlusal wall, up to the axial or pulpal wall and 3=dye penetration along the axial or pulpal wall. For each sample, the worst score was registered. The data were statistically analyzed by Kruskal-Wallis analysis of variance to determine any statistical significant differences in microleakage scores among the groups at a p-value of 0.05. Mann-Whitney U-test was

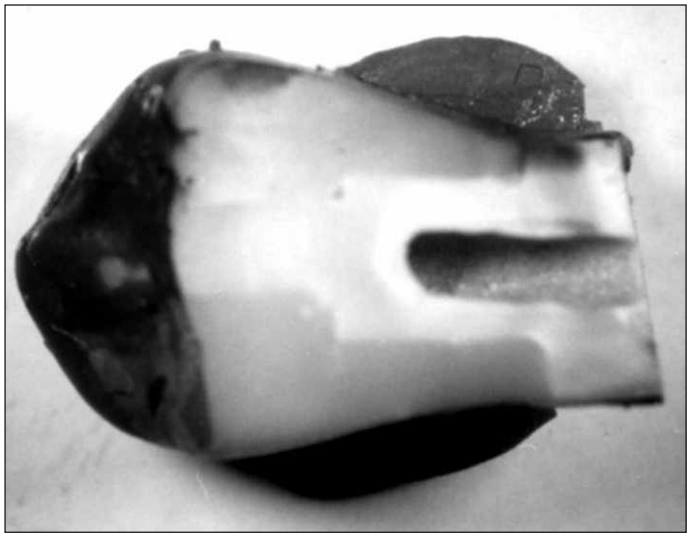


Figure 1. *Dye penetration under the increment with flowable composite and beyond 1/3 of the gingival wall (Score 2) (Group 2).*

performed to compare the groups with each other at the 0.05 significance level.

RESULTS

Occlusal and gingival microleakage scores and medians of the study groups are presented in Tables 2 and 3. Kruskal-Wallis analysis of variance indicated no significant difference among groups 1a, 2a and 3a (p=0.71). Similarly, there were no significant differences between groups 1b, 2b and 3b (p=0.22).

There were no statistically significant differences for occlusal microleakage scores between groups restored with or without flowable materials (p>0.05). However, flowable materials were found to reduce microleakage along the gingival walls of three resin composites (p<0.05).

Dye penetration at the gingival margin is shown in Figure 1.

DISCUSSION

Microleakage is the most significant disadvantage associated with the use of composite restorative materials. It is dependent upon several factors including adaptation of resin material to tooth surface, the bonding material used, the technique of bonding, polymerization shrinkage and the thermal stability of the material. Clinically, it is evident as staining around the margins of the restoration, post-operative sensitivity, secondary caries, restoration failure, pulpal pathology or pulpal death (Eick & Welch, 1986; Taylor & Lynch, 1993). Studies show that microleakage into dentin remains a significant problem (Davidson & Feilzer, 1997).

Restorative materials when tested *in vitro* fail to simulate the dynamic intra oral thermal changes induced by routine eating and drinking. Thermocycling is often employed in laboratory experiments to simulate stresses in the oral cavity. The absence of outward flow of dentinal fluid and completely altered dentinal surface fusion due to extraction lead to a poor correlation between *in vivo* and *in vitro* conditions (Pashley, 1990). However, microleakage studies can provide some initial information and comparison of different new restorative materials (Abdalla & Davidson, 1993).

Packable composites are one of the recently developed restorative materials. The main advantages of packable composites over hybrid resins are the elimination of stickiness of the material and the ability to produce good approximal contacts by condensing incrementally (Kelsey & others, 2000; Leevailoj & others, 2001; Leinfelder, Radz & Nash, 1998). Despite being more favorable, concerns related to their ability to sufficiently wet and adapt to cavity walls, especially at the cervical margins below the cemento-enamel junction, have been raised (Tyas, Jones & Rizkalla, 1998; Leevailoj & others, 2001). With the development of flowable composites, their placement under packable resins has been advocated in order to reduce microleakage at the cervical margins (Bayne & others, 1998; Leevailoj & others, 2001). The flowability and injectability of flowable composites make them very attractive when placing in difficult areas such as the proximal boxes of Class II restorations (Bayne & others, 1998; Beznos, 2001; Estafan & Estafan, 2000; Payne, 1999).

In vitro studies have reported significant effects of using flowable composites as liners and gingival increments in reducing the gingival microleakage of packable and microhybrid resin restorations (Leevailoj & others, 2001; Prager, 1997). Similar to these results, in this study, the utilization of flowable materials under both packable and microhybrid resins resulted in less gingival microleakage regardless of the material used. However, this study investigated the absolute effect of gingival increment with flowable materials in reducing gingival microleakage of Class II restorations. Therefore, flowable materials were not placed under packable and microhybrid resins. One of the disadvantages of flowable materials may be related to their stickiness. Flowable materials were also observed to pool over the gingival increment. This makes their manipulation difficult especially when a lining over a gingival increment was attempted.

Table 2: *Occlusal Microleakage Scores*

Group	Flowable	Medians	Microleakage Scores			
			0	1	2	3
1	Yes	0	11	3	1	0
	No	0	9	5	1	0
2	Yes	0	13	2	0	0
	No	0	11	3	1	0
3	Yes	0	9	4	1	1
	No	0	11	3	0	1

Table 3: *Gingival Microleakage Scores*

Group	Flowable	Medians	Microleakage Scores			
			0	1	2	3
1	Yes	0	11	3	1	0
	No	1	4	4	2	5
2	Yes	0	9	3	1	2
	No	2	0	1	7	7
3	Yes	0	12	2	0	1
	No	1	4	4	2	5

In contrast to the results of this study, flowable composites are expected to shrink more since they have less filler loading (Bayne & others, 1998). However, the elastic modulus of flowable composites range from 1 GPa to 5 GPa, which is lower than an average hybrid resin and dentin. The lower elastic modulus of a material indicates a greater ability to flex with the tooth to accommodate the inherent modulus of the tooth, which will eliminate gap formation and subsequent microleakage (Estafan & Estafan, 2000; Prager, 1997). Moreover, microleakage decreases as the seal between the tooth and resin increases, depending on the ability of the resin to adequately penetrate the tooth resin interface by capillary action. The viscosity and wettability has been known to influence capillary action (Craig, 1985). An additional advantage to lower elastic modulus of a resin is its ability to absorb shrinkage stress of the overlying high loaded resin by acting as a flexible intermediate layer (Unterbrink & Liebenberg, 1999; Estafan & Estafan, 2000; Prager, 1997; Rooklidge, Boyer & Bouschlicher, 1999; Kemp-Scholte & Davidson, 1990). These realities were supported by both this study and other *in vitro* studies in which flowable composites showed more or equal resistance to microleakage compared to microhybrid resins (Estafan and others, 1999; Estafan & Estafan, 2000; Ferdianakis, 1998). However, some studies found no difference in resistance to microleakage when flowable composite liners were placed with composite restorations (Walshaw & McComb, 1998; Malmstrom & others, 2002; Neme & others, 2002).

In this study, the microleakage scores of two packable resin composites with a microhybrid resin composite were also compared. Although packable composites require greater forces to condense and are more difficult to adapt to cavities than microhybrid composites (Leevailoj & others, 2001), the results of this study demonstrated no statistically significant differences among the materials used. However, placement of a packable composite is more attractive than a microhybrid since it does not stick to dental instruments.

Much of the current literature focuses on elimination of microleakage, which is one of the major factors determining the long-term success of restorations. Within the limitations of this study, it can be concluded that the use of a flowable composite or compomer as the first gingival increment of Class II restorations with both packable and microhybrid composites decreases gingival microleakage. However, further clinical research is needed to support the use of these materials.

CONCLUSIONS

The results obtained in this study indicate that the use of flowable materials–Dyract Flow, Filtek Flow and Tetric Flow–as gingival increments of Class II restorations with packable and microhybrid composites, SureFil, Filtek P60 and Tetric Ceram, decreased gingival microleakage.

(Received 11 February 2003)

References

3M Dental Products (1999) *Filtek P-60 Posterior Restorative System Technical Manual* St Paul MN 55144.

Abdalla AI & Davidson CL (1993) Comparison of the marginal integrity of *in vivo* and *in vitro* Class II composite restorations *Journal of Dentistry* **21(3)** 158-162.

Bayne SC, Thompson JY, Swift EJ Jr, Stamatiades P & Wilkerson M (1998) A characterization of first-generation flowable composites *Journal of the American Dental Association* **129(5)** 567-577.

Beznos C (2001) Microleakage at the cervical margin of composite Class II cavities with different restorative techniques *Operative Dentistry* **26(1)** 60-69.

Craig RG (1985) *Restorative Dental Materials* 7th ed St Louis Mosby 245-247.

Davidson CL & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer based restoratives *Journal of Dentistry* **25(6)** 435-440.

Dentsply De Trey (1998) *SureFil Technical Manuel High Density Posterior Restorative* Dentsply De Trey Konstanz.

Eick JD & Welch FH (1986) Polymerization shrinkage of posterior composite resins and its possible influence on post-operative sensitivity *Quintessence International* **17(2)** 103-111.

Estafan D, Schulman A & Calamia J (1999) Clinical effectiveness of a Class V flowable composite resin system *Compendium* **20(1)** 11-16.

Estafan AM & Estafan D (2000) Microleakage study of flowable camposite resin systems *Compendium* **21(9)** 705-714.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Ferrari M, Vichi A, Mannocchi F & Davidson CL (1998) Sealing ability of two "compomers" applied with and without phosphoric acid treatment for Class V restorations *in vivo Journal of Prosthetic Dentistry* **79(2)** 131-135.

Ferdianakis K (1998) Microleakage reduction from newer esthetic restorative materials in permanent molars *Journal of Clinical Pediatric Dentistry* **22(3)** 221-229.

Jackson RD & Morgan M (2000) the new posterior resins and a simplified placement technique *Journal of the American Dental Association* **131(3)** 375-383.

Kelsey WP, Latta MA, Shaddy RS & Stanislav CM (2000) Physical properties of three packable resin-composite restorative materials *Operative Dentistry* **25(4)** 331-335.

Kemp-Scholte CM & Davidson CL (1990) Complete marginal seal of Class V resin composite restorations effected by increased flexibility *Journal of Dental Research* **69(6)** 1240-1243.

Leevailoj C, Cochran MA, Matis BA, Moore BK & Platt JA (2001) Microleakage of posterior packable resin composites with and without flowable liners *Operative Dentistry* **26(3)** 302-307.

Leinfelder KF, Sluder TB, Santos JF & Wall JT (1980) Five- year clinical evaluation of anterior and posterior restorations of composite resin *Operative Dentistry* **5(2)** 57-65.

Leinfelder KF (1991) Using composite resin as a posterior restorative material *Journal of the American Dental Association* **122(4)** 65-70.

Leinfelder KF, Radz GM & Nash RW (1998) A report on a new condensable composite resin *Compendium* **19(3)** 230-237.

Malmstrom HS, Schlueter M, Roach T & Moss ME (2002) Effect of thickness of flowable resins on marginal leakage in Class II composite restorations *Operative Dentistry* **27(4)** 373-380.

Neme AM, Maxson BB, Pink FE & Aksu MN (2002) Microleakage of Class II packable resin composites lined with flowables: An *in vitro* study *Operative Dentistry* **27(6)** 600-605.

Opdam NJ, Roeters JJ, Peters TC, Burgersdijk RC & Teunis M (1996) Cavity wall adaptation and voids in adhesive Class I resin composite restorations *Dental Materials* **12(4)** 230-235.

Pashley DH (1990) Clinical considerations of microleakage *Journal of Endodontics* **16(2)** 70-77.

Payne JH (1999) The marginal seal of Class II restorations: Flowable composite resin compared to injectable glass ionomer *Journal of Clinical Pediatric Dentistry* **23(2)** 123-130.

Phillips RW, Avery DR, Mehra R, Swartz ML & Cune RJ (1973) Observations on a composite resin for Class II restorations: Three year report *Journal of Prosthetic Dentistry* **30(6)** 891-897.

Prager MC (1997) Using flowable composites in direct posterior restorations *Dentistry Today* **16(7)** 62-69.

Rooklidge E, Boyer D & Bouschlicher M (1999) Cusp deformation by shrinkage of condensable composite *Journal of Dental Research* **78** Abstract #2349 p 399.

Taylor MJ & Lynch E (1993) Marginal adaptation *Journal of Dentistry* **21(5)** 265-273.

Tyas MJ, Jones DW & Rizkalla AS (1998) The evaluation of resin composite consistency *Dental Materials* **14(6)** 424-428.

Tung FF, Estefan D & Scherer W (2000) Microleakage of a condensable resin composite: An *in vitro* investigation *Quintessence International* **31(6)** 430-434.

Unterbrink GL & Liebenberg WH (1999) Flowable resin composites as "filled adhesives:" Literature review and clinical recommendations *Quintessence International* **30(4)** 249-257.

Unterbrink GL & Muessner R (1995) Influence of light intensity on two restorative systems *Journal of Dentistry* **23(3)** 183-189.

Verluis A, Tantbirojn D & Douglas WH (1998) Do dental composites always shrink toward the light? *Journal of Dental Research* **77(6)** 1435-1445.

Walshaw PR & McComb D (1998) Microleakage in Class II resin composites with low-modulus *Journal of Dental Research* **77** Abstract #204 p 131.

©Operative Dentistry, 2004, **29-2,** 168-175

Shear Bond Stability of Current Adhesive Systems to Enamel

H Wang • Y Shimada • J Tagami

Clinical Relevance

Extending storage time and shear bond strength to enamel of the self-etching primer system and single-bottle adhesive system were decreased.

SUMMARY

This study evaluated the micro-shear bond strength of two commercially available resin bonding systems (Single Bond, 3M, USA and SE Bond, Kuraray, Japan) to human enamel. One hundred and twenty enamel sections were prepared from extracted non-carious human molars by cutting with a slowly rotating blade. The enamel surfaces were polished with #280-grift SiC paper under running water. These surfaces were randomly divided into two groups and treated with either Single Bond or Clearfil SE Bond according to manufacturers' instructions. After the bonding procedures, a micro tygon tubing with an internal diameter approximately 0.7 mm and 0.5 mm in height was placed on the enamel surface and Clearfil AP-X resin filled the tube. After the resin was photo-irradiated, the tygon tube was removed. The specimens were further divided into 12 groups according to storage time. All specimens were stored in isotonic sodium chloride solution at 37°C. The bond strength was then measured by means of micro-shear bond testing at one day, one week, one month, three months, six months and one year. After testing, the fractured surfaces and interfaces were observed using scanning electron microscopy (SEM). The data was analyzed by two-way ANOVA and Fisher's PLSD tests at 95% level of confidence. The result was that both factors (storage time and material) affected bond strength and there was a statistically significant interaction between them. In general, bond strength decreased with time for both materials. The highest bond strengths were achieved at one day with SE Bond and Single Bond. For Single Bond, from one month to one year, the bond strength dramatically decreased. For SE Bond, the decrease in bond strength was gradual. After one-year storage, the bond strength drastically decreased and this value was not significantly different from the results of the one-year storage of Single Bond.

*Hao Wang, DDS, graduate student, Cariology and Operative Dentistry, Department of Restorative Sciences, Graduate School, Tokyo Medical and Dental University, Tokyo, Japan

Yasushi Shimada, DDS, PhD, instructor, Tokyo Medical and Dental University, Tokyo, Japan

Junji Tagami, DDS, PhD, professor and chair, Tokyo Medical and Dental University, Tokyo, Japan

*Reprint request: 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549l; e-mail: wangope@tmd.ac.jp

INTRODUCTION

In 1955, Buonocore introduced the so-called acid-etch technique that enables the bonding of resin composites to the enamel surface (Buonocore, 1955). Since then, the development of adhesive resin has changed the design of cavity preparations, replacing the extensive removal of tooth structure with more conservative preparations (Buonocore, 1975). Recently, although many commercial adhesive resin systems are available, there are two major simplified approaches to producing good hybridization and adequate bonds (Nakabayashi, Kojima & Masuhara, 1982). The first is the total etching technique; commercially available etchants contain 30% to 40% phosphoric acid that provides enamel surfaces with the most retentive surface. Acid etching creates a porous enamel surface layer ranging in depth from 5 to 50 µm (Silverstone & others, 1975; Hannig, Reinhardt & Bott, 1999). A low-viscosity bonding agent is used to penetrate the microporosities created in the enamel surface. After polymerization of the resin bonding agent, a durable attachment to the enamel is achieved by micromechanical retention (Silverstone & others, 1975; Kanemura, Sano & Tagami, 1999; Shinchi, Soma & Nakabayashi, 2000; Shimada & others, 1999, 2002; Shimada & Tagami, 2003). The second approach is the self-etching-priming technique that simultaneously conditions both enamel and dentin using an acidic primer. With this process there is no need to rinse off reaction products or residual phosphoric acid ester, because both are subsequently polymerized into the bonding layer (Hannig & others, 1999). Both approaches have led to an increase in bond strength. Although the majority of bond strength tests were performed using enamel extracted from human and bovine teeth, very few studies are available that evaluate the micro-shear bond strength of human enamel over time using different forms of enamel conditioning. Additionally, there is little or no agreement in bond strength data among different laboratories. In this study, the authors investigated the durability of micro-shear bond strengths to human enamel of two commercially available resin bonding systems (Clearfil SE Bond and Single Bond) and observed the morphological changes in fracture interface by scanning electron microscopy.

Table 1: *Materials Used*

Materials	Composition
Clearfil SE Bond	
Primer	MDP, HEMA, Hydrophilic dimethacrylate, dl-Camphorquinone, Aromatic tert-amine, Water
Bond Liquid	MDP, Bis-GMA, HEMA, Hydrophilic dimethacrylate, Photo initiator, Aromatic tert-amine, Silanated colloidal silica
Single Bond (#19981008)	
Etchant	35%Phosphoric acid
Adhesive	HEMA, Bis-GMA, Dimethacrylates, Metacrylate, Functional copolymer, (polyacrylic and polyitaconic acids), Photoinitiator, ethanol, Water
Clearfil AP-X (#01251)	Light-curing hybrid resin composite

Table 2: *Application Techniques*

Single Bond		Clearfil SE Bond	
(1) Apply etchant	15 seconds	(1) Apply primer	20 seconds
(2) Rinse	15 seconds	(2)Dry	-
(3) Blot dry with absorbent paper	-	(3) Apply adhesive	-
(4) Apply two coats of adhesive	-	(4) Apply adhesive	-
(5) Apply light cure	10 seconds	Apply light cure	10 seconds

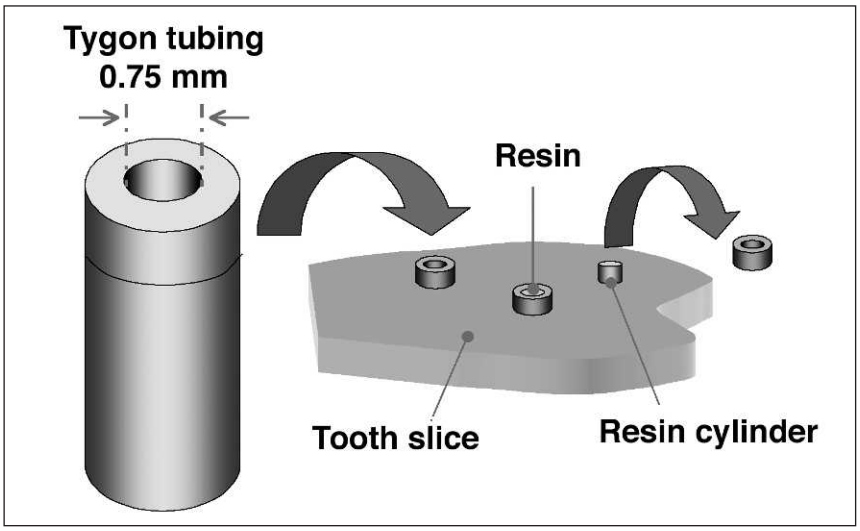


Figure 1: Specimen preparation.

METHODS AND MATERIALS

Table 1 lists the materials and composition for this study.

Specimen Preparation

One hundred and twenty enamel sections were prepared from extracted non-carious human molars by

cutting the proximal surfaces with a slow rotating blade. The enamel surfaces were polished with #280-grit silicon carbide paper under running water. The teeth were randomly divided into two groups and treated with Clearfil SE Bond (Kuraray Medical Inc, Tokyo, Japan) or Single Bond (3M, St Paul, MN, USA) according to manufacturers' instructions (Table 2). Single Bond was applied using the wet-bonding technique. After the bonding procedure, micro tygon tubing with an internal diameter of approximately 0.7 mm and 0.5 mm in height was placed on the enamel surface and Clearfil AP-X resin (Kuraray Medical Inc, Tokyo, Japan) filled the tube. After the resin was light cured for 40 seconds with a quartz–tungsten halogen lamp (Optilux, Demetron Research Corporation/Sybron Dental Specialties, Orange, CA, USA), the tygon tube was removed (Figure 1).

The specimens were further divided into 12 groups according to storage time: one day, one week, one month, three months, six months and 12 months. All specimens were stored in isotonic sodium chloride solution at 37°C.

Micro-shear Bond Test

After storage, the specimens were tested for micro-shear bond strength (Figure 2) using the EZ test tensile apparatus (Shimazu CO, Kyoto, Japan). Before the test, all samples were checked under an optical microscope (30x) for bonding defects. The five samples that showed apparent interfacial gap formation, bubble inclusion or some other defects were excluded from this study, and the five samples were supplemented with no bonding defects to ensure 10 samples in each group. The results of the micro-shear bond strength test were statistically analyzed using two-way ANOVA and Fisher's PLSD test.

Scanning Electron Microscopy Observation

After testing, all the fractured surfaces were visually inspected to determine the mode of fracture according to fracture position. The fractured specimens were classified into one of four categories as follows: A-100% adhesive failure between the enamel or hybrid enamel layer and overlying adhesive resin; B-Adhesive failure between the enamel or hybrid enamel layer and overlying adhesive resin combined with cohesive failure in enamel (adhesive failure in more than 50% of the debonded zone); C-Cohesive failure in enamel combined with adhesive failure between the enamel or hybrid enamel layer and overlying adhesive resin (cohesive failure in enamel in more than 50% of the debonded zone); D-100% cohesive failure in enamel (Table 3). In addition, representative samples were also observed using a scanning electron microscope (SEM, JSM-5310LV CO, Tokyo, Japan) to confirm the accuracy of the visual inspection. The results were statistically analyzed using the Kruskal-Wallis test ($p>0.05$).

For scanning electron microscopy of the interface, the specimens that had been bonded with Clearfil SE Bond

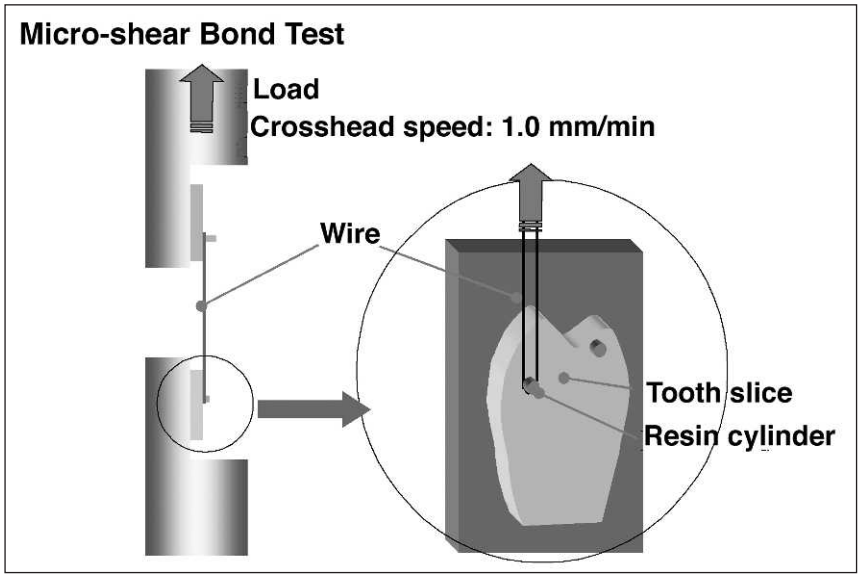


Figure 2: Micro shear bond test.

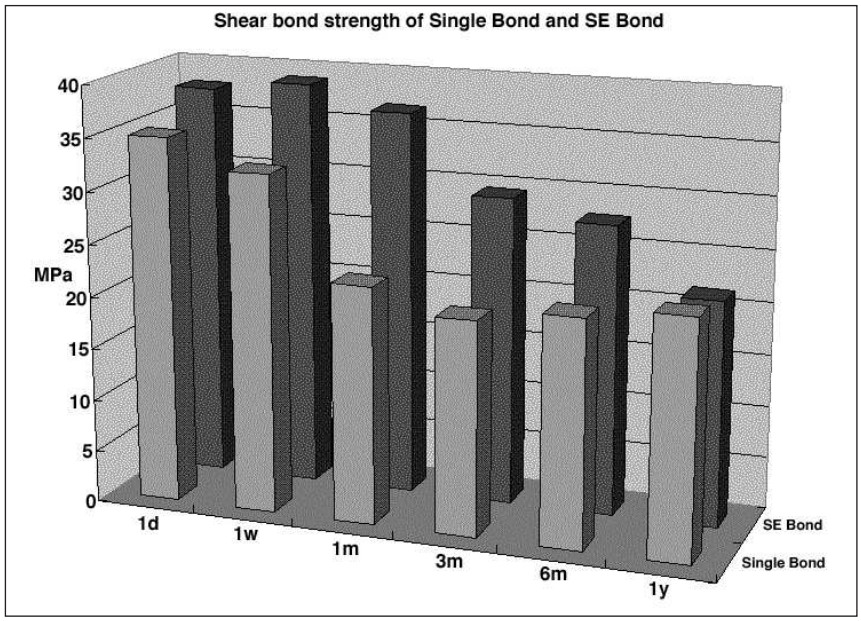


Figure 3. Shear bond strength of Single Bond and SE Bond.

and Single Bond were ground and polished using wet silicon carbide papers and diamond pastes of decreasing abrasiveness down to 0.25 µm. The specimens were then subjected to argon ion etching (E1S-1E, ELionix Ltd, Tokyo, Japan) for seven minutes. The operating conditions for the ion source were kept at a constant anode voltage of 1KV and ion current density of 0.2 mA/cm^2, with the ion beam directed at 90°C to the specimen surface. These specimens were then sputter-coated with gold and observed by SEM.

RESULTS

Micro-shear Bond Test

Table 3 and Figure 3 show the micro-shear bond strength values of Clearfil SE Bond and Single Bond to human enamel. Both factors (s torage time and material) affected the bond strength and there was a statistically significant interaction between them. In general, bond strength decreased with time for both materials. The highest bond strengths were achieved at one day with SE Bond and Single Bond. For Single Bond, both the strength of one day and one week did not show any difference. However, from one month to one year, the bond strength dramatically decreased. For SE Bond, the decrease in bond strength was gradual. The highest values were obtained at one day and the bond strength decreased slightly up to one month but was not statistically significant. At three months, the bond strength significantly decreased and there was no difference between three and six months results. After one-year storage, the bond strength drastically decreased and this value was not significantly different from the results of one-year storage for Single Bond (22.7MPa).

Scanning Electron Microscopy Observation

Table 3 illustrates the mode of fracture exhibited for each group as the time varied. The failure modes for SE Bond specimens were various patterns; a complex failure pattern occurred and cohesive failure mixed with adhesive failure. For Single Bond, the fracture modes were mainly classified as adhesive failure (Figure 4). However, no significant differences in mode of failure were found among the adhesive systems or the test periods (Kruskal-Wallis test, $p>0.05$). The SEM observations of the resin-enamel interface are shown in Figures 5 and 6. The authors observed apparent changes in the adhesive layer, along with the duration extending for Single Bond, especially after one-year storage.

DISCUSSION

In this study, the authors investigated the shear bond long-term durability of two commercially available resin bonding systems SE Bond and Single Bond. Most of the currently marketed adhesives achieve high bond strength immediately after polymerization of the resin (Burrow & others, 1994; Hannig & others, 1999). However, the longevity of adhesive bond is still one of the areas of interest in adhesive dentistry. Several

Figure 4: ***SEM observations of debonded sites after the micro-shear bond test.***

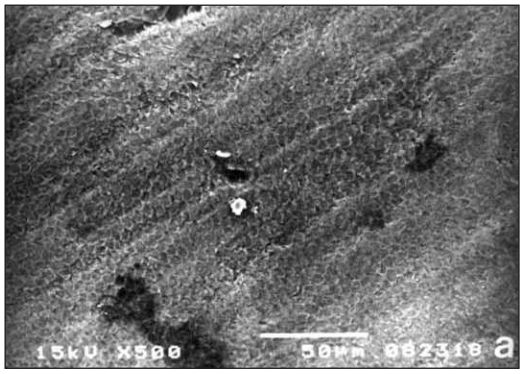


Figure 4*a. Adhesive failure for Single Bond specimen.*

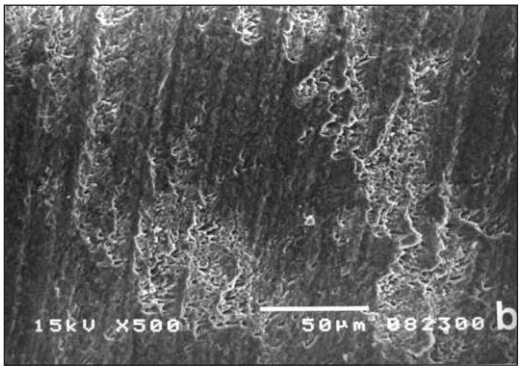


Figure 4*b. Complex failure for SE Bond specimen.*

Table 3: *Shear Bond Strength (Mpa) and Mode of Failure*

			Single Bond	SE Bond
	Single Bond	**SE Bond**	**A B C D**	**A B C D**
1 day	35.0±6.68 a	37.9±8.22 a,b	9 1 0 0	5 3 2 0
1 week	32.3±9.08 a	38.9±5.06 b	8 2 0 0	8 1 0 1
1 month	22.7±3.20 c	36.8±4.52 b	9 1 0 0	6 3 0 1
3 months	20.5±5.99 c	29.5±6.14 d	10 0 0 0	8 1 1 0
6 months	21.7±4.39 c	27.8±6.71 d	3 7 0 0	5 4 0 1
1 year	22.7±8.40 c	21.6±4.76 c	6 2 1 1	6 3 1 0

Mean ± SD, n=10, different letters indicate statistically significant difference.

studies have been done related to the durability of adhesive systems for dentin (Chan, Reinhardt & Boyer, 1985; Kato & Nakabayashi, 1998; Kiyomura, 1987; Burrow, Inokoshi & Tagami, 1999; Gwinnett & Yu, 1995; Okuda & others, 2002). Several studies related to durability are often performed using either thermal cycling or load stress (Miyazaki, Sato & Onose, 2000). This can be regarded as a means of accelerating aging of the bonding system, which is somewhat similar to that proposed for aging of resin composites at 60°C (Asmussen, 1981). Although these evaluation methods are adequate for determining the adhesive strength of the material, they do not provide any information about the durability of the material from a clinical long-term viewpoint. Because such factors as water absorption into resin, hydrolysis and those factors related only to time cannot be produced in short-term tests lasting only a few days (Braden, Causton & Clarke, 1976; Burrow, Tagami & Hosoda, 1993), the use of thermal cycling to simulate durability does not seem similar to the long-term storage presented in this study.

The results of this study showed that Clearfil SE Bond and Single Bond produced high bond strengths to normal enamel at the first day after restoration. These data also agree with Hannig & others (1999), who reported that self-etching primers could provide an effective alternative to conventional phosphoric acid etchants in conditioning the enamel surface to secure a durable bonding; however, the bond strength gradually decreased with time.

In the case of the Single Bond system, it created a stable resin tag and mechanical retention between enamel and resin composite, which were solely composed of cured resin (Soetopo, Beech & Hardwick, 1978). However, because resin does not completely infiltrate etched enamel (Shinchi & others, 2000), a region of unprotected enamel prisms might be susceptible to hydrolytic degradation after long-term storage in isotonic sodium chloride solution. In addition, after long-term storage in saline, the authors observed apparent changes in the adhesive layer, especially in one year storage of Single Bond (Figure 5). This might be the result of the absorption of a significant amount of water by resin bonds (Burrow & others, 1999). Presumably, it might cause changes in the bonding interface after long-term storage, such as hydrolysis of adhesive resin (Shono & others, 1999) and, as a consequence, cause a decrease in micro-shear bond strength. The majority of the failure of Single Bond was classified as adhesive failure, and only one case of cohesive failure in enamel was observed in the one-year group. It is highly likely that the fracture toughness of adhesive may be decreased so that the mode of failure is mostly adhesive failure, even though the bonding obtained is decreased. This will affect the longevity of adhesive bond. It is also possible that etched enamel caused the degradation, which might affect the lower bonding. During long-term storage in saline, the apatite in etched enamel would be dissolved due to the common ion effects of calcium and phosphate (Yoshiyama & others, 1996).

The Clearfil SE Bond system contains a self-etching primer and a bonding agent. Unlike previous versions, resin monomer components, photoinitiator and accelerator are combined into a single bottle; the simulta-

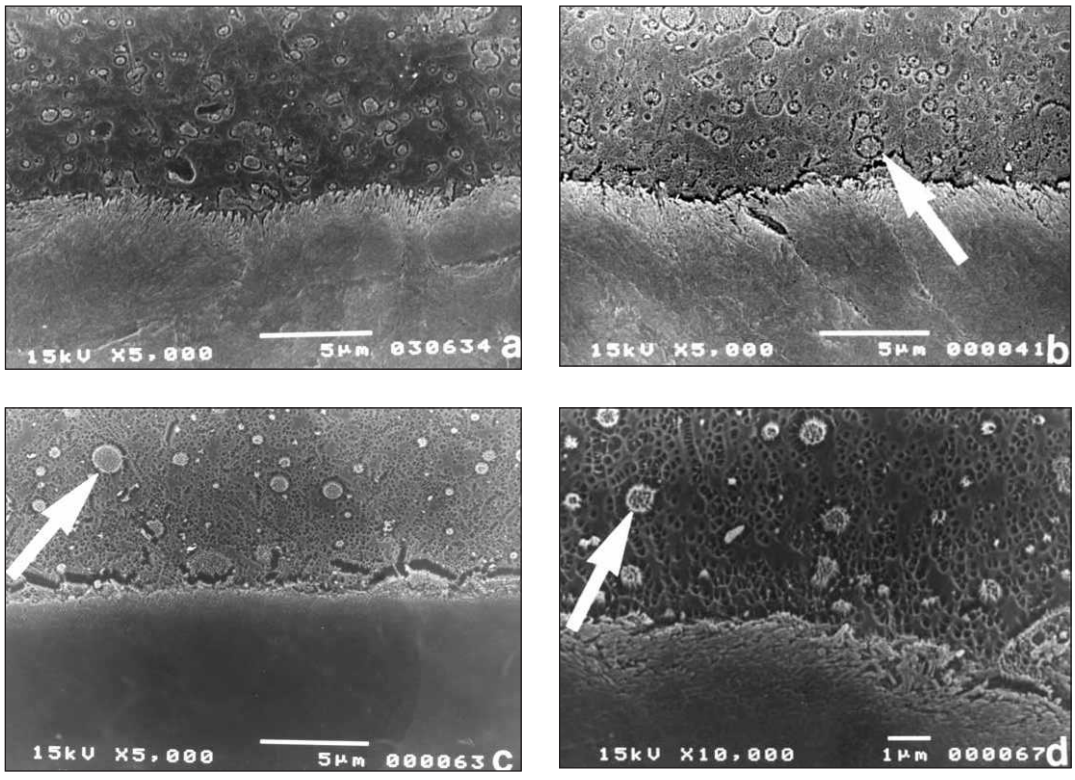


Figure 5. *SEM observations of the resin-enamel interfaces of Single Bond for the different terms (a) one day, (b) six months, (c) one year and (d) one year at higher magnification. The apparent changes in the adhesive layer, especially in the one-year group, were observed. Note the presence of circular amorphous hybrid layer-like structures (arrow) inside the adhesive layer. These structures decreased and bubble-like structures appeared in one-year group.*

neous etching and priming facilitates penetration of the adhesive resin monomer into etched enamel (Perdigão & others, 1997).

However, the solo application of a self-etching primer resulted in a shallow etching pattern that might be the result of mild decalcification. It may occur because etching might cease gradually due to acidity of the primer being neutralized on the enamel surface (Itou & others, 1994; Pashley & Carvalho, 1997), or it may be a result of the self-etching primer selectively decalcifying the interprismatic region due to weak acidity and partly decalcifying the wall of the prism core (Torii & others, 2002). After long-term storage, the authors observed small cracks and porosities between the enamel and adhesive in the SEM observation (Figure 6). This might have occurred because resin tag formation is relatively shallow and the change in mechanical properties deteriorates after storage in isotonic sodium chloride solution. Clearfil SE Bond presented various failure patterns but was mainly classified as adhesive failure. In the Clearfil SE Bond bonding agent formulation, there are microfillers. Because of this, the decrease in shear bond strength of Clearfil SE Bond may be slower than Single Bond after long-term storage.

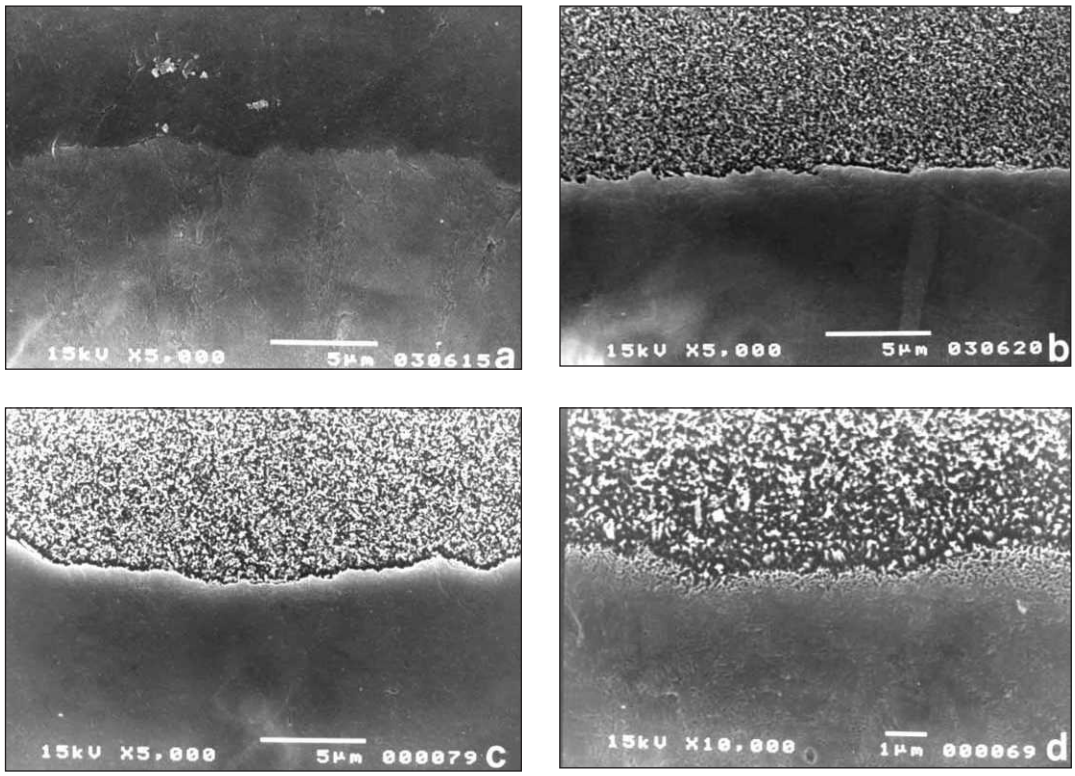


Figure 6. *SEM observations of the resin-enamel interfaces of SE Bond for the different terms (a) one day, (b) six months, (c) one year and (d) one year at higher magnification. Small cracks and porosities between the adhesive and enamel were observed.*

Many factors can affect the stability of adhesive systems' performance on enamel. It has been reported that the hardness of enamel decreases after storage in physiological saline due to the loss of surface calcium and the softening effect of distilled water is less than that of saline (Mühlemann, 1964). In addition, from the aspect of the resin composite material, water diffusion into the bonding interface between the adhesive and tooth surface was found to cause resin to swell and become plasticized (Söderholm, 1991). Evaluating bonding stability is essential since the bond between the restoration and tooth substrate is clinically significant only if it is long-lasting. Additionally, if load stress or thermolcycling was added, bond strength would further decrease (Miyazaki & others, 2000). One study reported the marginal degradation or discoloration of resin composite restorations *in vivo* (Van Dijken, 1986). The decreased bonding of enamel observed in this study would partially be attributed to the defective margins of long-lasting restorations.

Other studies reported a marked decrease in dentin bond strength after one year (Okuda & others, 2001; 2002). This decrease in dentin bond strength seemed to be more significant than in enamel. It implies that the longevity of resin-enamel bonds is longer than resin-dentin bonds in clinic. Also, further areas of research that should be considered from the results of this study include evaluating the stability of current adhesive systems on enamel in different oral environmental conditions. In the case of both systems, bonding was decreased from 35 or 38 MPa to 21 or 22 MPa (Single Bond and Clearfil SE Bond, respectively) in one year. The authors speculate that these bond strength values would decrease more if the storage time in saline were extended. Consequently, it is still not clear whether bond strength around 21 MPa is sufficient for bonding in clinic.

CONCLUSIONS

It was concluded that the bond strength to enamel of both materials decreased significantly with time when stored in isotonic sodium chloride solution. The one-day strengths of the two materials were not statistically different. The same was true when comparing the one-year bond strengths.

(Received 11 February 2003)

References

Asmussen E (1981) An accelerated test for color stability of restorative resins *Acta Odontologica Scandinavia* **39(6)** 329-332.

Buonocore MG (1955) A simple method of increasing adhesion of acrylic filling materials to enamel surfaces *Journal of Dental Research* **34(6)** 849-853.

Buonocore MG (1975) *The Use of Adhesive in Dentistry* Springfield IL Thomas.

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

Burrow MF, Tagami J & Hosoda H (1993) The long term durability of bond strengths to dentin *The Bulletin of Tokyo Medical and Dental University* **40(4)** 173-191.

Burrow MF, Tagami J, Negishi T, Nikaido T & Hosoda H (1994) Early tensile bond strengths of several enamel and dentin bonding systems *Journal of Dental Research* **73(2)** 522-528.

Burrow MF, Inokoshi S & Tagami J (1999) Water sorption of several bonding resins *American Journal of Dentistry* **12(6)** 295-298.

Chan DC, Reinhardt JW & Boyer DB (1985) Composite resin compatibility and bond longevity of a dentin bonding agent *Journal of Dental Research* **64(12)** 1402-1404.

Gwinnett AJ & Yu S (1995) Effect of long-term water storage on dentin bonding *American Journal of Dentistry* **8(2)** 109-111.

Hannig M, Reinhardt K-J & Bott B (1999) Self-etching primer vs phosphoric acid: An alternative concept for composite-to-enamel bonding *Operative Dentistry* **24(3)** 172-180.

Itou k, Torii Y, Suzuki K & Inoue K (1994) Adhesion of restorative resin to tooth-adhesion promoted by Liner Bond II *Adhesive Dentistry* **12** 174-181.

Kanemura N, Sano H & Tagami J (1999) Tensile bond strength to and SEM evaluation of ground and intact enamel surfaces *Journal of Dentistry* **27(7)** 523-530.

Kato G & Nakabayashi N (1998) The durability of adhesion to phosphoric acid etched, wet dentin substrates *Dental Materials* **14(5)** 347-352.

Kiyomura M (1987) Bonding strength to bovine dentin with 4-META/MMA-TBB resin: Long term stability and influence of water *The Journal of the Japanese Society for Dental Materials and Devices* **6(6)** 860-872.

Miyazaki M, Sato M & Onose H (2000) Durability of enamel bond strength of simplified bonding systems *Operative Dentistry* **25(2)** 75-80.

Mühlemann HR (1964) Storage medium and enamel hardness *Helvetica Odontologica Acta* **8** 112-117.

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.

Okuda M, Pereira PN, Nakajima M & Tagami J (2001) Relationship between nanoleakage and long-term durability of dentin bonds *Operative Dentistry* **26(5)** 482-490.

Okuda M, Pereira PN, Nakajima M, Tagami J & Pashley DH (2002) Long-term durability of resin dentin interface: Nanoleakage vs microtensile bond strength *Operative Dentistry* **27(3)** 289-296.

Pashley DH & Carvalho RM (1997) Dentine permeability and dentine adhesion *Journal of Dentistry* **25(5)** 355-372.

Perdigão J, Lopes L, Lambrechts SP, Leitão J, Van Meerbeek B & Vanherle G (1997) Effects of a self-etching primer on enamel shear bond strengths and SEM morphology *American Journal of Dentistry* **10(3)** 141-146.

Shimada Y, Antonucci JM, Schumacher GE, McDouough WG & Tagami J (1999) Effects of regional tooth structure and sectioning orientation on micro-shear bond strength. Tagami J, Toledano M & Prati C ed A*dvanced Adhesive Dentistry* 3rd International Kuraray Symposium Kuraray Co Ltd 91-103.

Shimada Y, Senawongse P, Harnirattisai C, Burrow MF, Nakaoki Y & Tagami J (2002) Bond strength of two adhesive systems to primary and permanent enamel *Operative Dentistry* **27(4)** 403-409.

Shimada Y & Tagami J (2003) Effects of regional enamel and prism orientation on resin bonding *Operative Dentistry* **28(1)** 20-27.

Shinchi MJ, Soma K & Nakabayashi N (2000) The effect of phosphoric acid concentration on resin tag length and bond strength of a photo-cured resin to acid-etched enamel *Dental Materials* **16(5)** 324-329.

Shono Y, Terashita M, Shimada J, Kozono Y, Carvalho RM, Russell CM & Pashley DH (1999) Durability of resin-dentin bonds *The Journal of Adhesive Dentistry* **1(3)** 211-218.

Silverstone LM, Saxton CA, Dogon IL & Fejerskov O (1975) Variation in the pattern of acid etching of human dental enamel examined by scanning electron microscopy *Caries Research* **9(5)** 373-387.

Soetopo, Beech DR & Hardwick JL (1978) Mechanism of adhesion of polymers to acid-etched enamel. Effect of acid concentration and washing on bond strength *Journal of Oral Rehabilitation* **5(1)** 69-80.

Söderholm KJ (1991) Correlation of *in vivo* and *in vitro* performance of adhesive restorative materials: A report of the ASC MD 156 task group on test methods for the adhesion of restorative materials *Dental Materials* **7(2)** 74-83.

Torii Y, Itou K, Hikasa R, Iwata S & Nishitani Y (2002) Enamel tensile bond strength and morphology of resin-enamel interface created by acid etching system with or without moisture and self-etching priming system *Journal of Oral Rehabilitation* **29(6)** 528-533.

Van Dijken JW (1986) A clinical evaluation of anterior conventional microfiller and hybrid composite resin fillings. A six-year follow-up study *Acta Odontologica Scandinavia* **44(6)** 357-367.

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, 2004, **29-2,** 176-181

Bond Strength of a Self-etching Adhesive System to Caries-Affected Dentin

AR Yazici • T Akca
G Özgünaltay • B Dayangaç

Clinical Relevance

The bond strength of a self-etching adhesive (Clearfil SE Bond) was greater in relation to sound dentin than to caries-affected dentin. Use of an additional acid etchant prior to applying Clearfil SE Bond did not have a positive effect on the bond strength of either sound or caries-affected dentin.

SUMMARY

This *in vitro* study evaluated the microtensile bond strengths of sound versus caries-affected dentin using a self-etching adhesive system, Clearfil SE Bond, with or without additional acid pre-conditioning.

Extracted human mandibular molars with occlusal caries extending halfway through the dentin were used. In the first group, the teeth were bonded with the self-etching adhesive Clearfil SE Bond according to the manufacturer's instructions. In the second group, prepared dentin surfaces were etched with 37% phosphoric acid prior to applying the same self-etching adhesive. After the bonding procedure, all specimens were built up with composite resin and stored in water for 24 hours. The teeth were serially sectioned vertically into 0.7-mm slabs and trimmed into an hourglass shape for measuring microtensile bond strength. Each specimen was attached to a Bencor device and stressed in tension at a crosshead speed of 1 mm/minute. Statistical analysis was performed using two-way ANOVA and the Tukey HSD test ($p<0.05$).

The microtensile bond strengths of Clearfil SE Bond to sound dentin (32.9) were significantly higher than to caries-affected dentin (15.9). In the second group where acid etching was performed prior to applying Clearfil SE Bond, there were no statistically significant differences between the microtensile bond strengths of sound (19.2) and caries-affected dentin (16.3). While bond strengths to sound dentin were decreased by using additional acid etching prior to applying

*A Rüya Yazici, DDS, PhD, teaching fellow, Hacettepe University, Faculty of Dentistry, Department of Conservative Dentistry, Ankara, Turkey

Taner Akca, DDS, PhD, research assistant, Hacettepe University, Faculty of Dentistry, Department of Pediatric Dentistry, Ankara, Turkey

Gül Özgünaltay, DDS, PhD, associate professor, Hacettepe University, Faculty of Dentistry, Department of Conservative Dentistry, Ankara, Turkey

Berrin Dayangaç, DDS, PhD, professor, Hacettepe University, Faculty of Dentistry, Department of Conservative Dentistry, Ankara, Turkey

*Reprint request: 06100, Sihhiye, Ankara, Turkey: e-mail: ruyay@hacettepe.edu.tr

Clearfil SE Bond, this procedure revealed no statistically significant differences in bond strengths for the caries-affected dentin.

INTRODUCTION

Self-etching adhesives have been introduced in an attempt to simplify the application of dentin adhesive systems. Most common self-etching adhesive systems involve two application steps with an acidic self-etching primer followed by the application of a resin overlayer, resulting in two-step self-etch adhesives (Van Meerbeek & others, 2001). Acidic monomers in the primer remove or modify the smear layer, superficially demineralize the dentin surface and simultaneously infiltrate into the demineralized space. As the pH value of self-etching/priming solution is generally low enough to demineralize the smear layer and the underlying dentinal surface, etching and priming of the cavity can be accomplished simultaneously (Watanabe, Nakabayashi & Pashley, 1994; Pashley & Carvalho, 1997). These systems do not require separate etching, rinsing or drying steps; therefore, the risk of over-etching and over-drying is eliminated and operating time is reduced (Van Meerbeek & others, 2001; Haller, 2000). However, in certain conditions, self-etching primers might not be able to penetrate through the smear layer. The acidity of the primer might be buffered by the smear layer, thereby reducing the potential for primer penetration. The use of additional acid has been suggested as a means of improving the bonding of self-etching adhesives to sound dentin, especially when there is a thick smear layer (Miyasaka & Nakabayashi, 1999).

Restorative materials usually bond to caries-affected dentin after the removal of carious tissue. However, bond strength tests of restorative materials are generally performed on sound dentin. Although the results are very useful with regard to comparing the effectiveness of adhesive systems, flat, sound dentin is not the substrate most regularly encountered in clinical situations.

It has been reported that self-etching adhesive systems show adequate bond strengths to sound dentin but little is known about their bond strength values for caries-affected dentin. Nakajima and others (1995) have reported that caries-affected dentin has lower bond strength than sound dentin for some bonding systems. Alternatively, an additional acid can be applied prior to applying the self-etching adhesive. It would be of interest to evaluate bond strengths to sound and caries-affected dentin.

This study compared the microtensile bond strength of a self-etching adhesive to caries-affected and sound human dentin and assessed the effect of acid preconditioning prior to applying the self-etching adhesive on bond strength values. The null hypotheses advanced in this study were (a) that the bond strength of a self-etching adhesive system (Clearfil SE Bond) to caries-affected dentin is lower than that to sound dentin and (b) that additional acid improves the bond strengths of this self-etching adhesive both to caries-affected and sound dentin.

METHODS AND MATERIALS

Twelve extracted human mandibular molars with occlusal caries extending approximately halfway through the dentin were used. The teeth were stored in 0.5% Chloramine T solution at 4°C and used within one month of extraction. After the coronal enamel was removed, the surfaces were wet ground on a model trimmer to expose a flat dentin surface with the carious lesion in the middle. The carious lesion was removed with conventional rotary instruments until all carious tissue had been excavated. Further grinding was performed with 600-grit SiC paper under running water to expose a flat surface according to the combined criteria of hardness to a sharp excavator, visual examination and staining with caries-detector dye (Kuraray Co, Ltd, Osaka, Japan). That is, the dentin was hard to an excavator and no longer stained bright red with the caries-detector dye. The teeth were randomly divided into two groups of six each. In the first group, a self-etching adhesive, Clearfil SE Bond (Kuraray Co), was applied on the prepared dentin surfaces by strictly following the manufacturer's instructions (Table 1). In the second group, the prepared dentin surfaces were first etched with 35% phosphoric acid gel (3M Dental Products, St Paul, MN, USA) for 15 seconds, then rinsed for 15 seconds and gently air-dried. Clearfil SE Bond was then applied as in the first group.

Following adhesive application, resin composite (Z100, 3M) was built up in three 1.5-mm-thick layers on the bonded surface. Each layer was light-cured for 40 seconds with a Hilux Expert (Benlioglu Dental, Ankara, Turkey) device with a light output not less than 550 mW/cm^2 as measured with a dental radiometer (Model 100, Demetron/Kerr, Danbury, CT, USA). The roots were removed from the crown approximately 2 mm below the cementoenamel junction using a low-speed diamond saw (Isomet, Buehler Ltd, Lake Bluff, IL, USA) under copious water spray. After removing the pulp of each tooth, the pulp chamber was acid etched with phosphoric acid for 15 seconds and filled with Z100 composite using the Single Bond (3M) adhesive system. This procedure reinforced the dentin near the pulp horns, where dentin was thinner.

Specimens were stored in water at 37°C for 24 hours, then vertically sectioned into 0.7-mm thick serial slabs perpendicular to the bonded surfaces of each tooth using a low-speed diamond saw (Isomet) under water-cooling (Pashley & others, 1999) (Figure 1). The slabs

were examined under a dissecting microscope (20x) to ensure that they were free of microcracks. The slabs in the two groups with and without acid etching prior to Clearfil SE Bond application were divided into two subgroups, one for caries-affected and the one for sound dentin. The four subgroups included four slabs from each tooth (two for sound and two for caries-affected dentin) which were selected visually. They were trimmed and shaped into an hourglass to create a bonding surface area of approximately 1 mm^2 using a super-fine diamond point. Ten to 12 slabs were selected according to the location of the pulp chamber to achieve estimated tubule direction to the bonded interface. They were then attached to the Bencor Multi-T apparatus (Danville, Engineering Co, Danville, CA, USA) and stressed in tension using a universal testing machine (Instron Corp, Canton, MA, USA) at a crosshead speed of 1 mm/minute (Sano & others, 1994). Microtensile bond strengths were determined by computing the ratio of maximum load divided by the bonded surface area. The results were analyzed using two-way ANOVA (sound vs caries-affected dentin with and without acid pretreatment) and multiple comparisons were made using the Tukey HSD test at α=0.05. The statistical analysis was performed using a software package (SPSS 8.0 for Windows, SPSS Inc, Chicago, IL, USA).

Table 1: *Adhesive System Used in This Study*

Adhesive System	Composition	Application
Clearfil SE Bond Kuraray Co, Ltd, Osaka, Japan	Primer: MDP, HEMA, hydrophilic dimethacrylate, CQ, N, N-Diethanol-p-toluidine, water Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, CQ, N, N-Diethanol- p-toluidine, silanated colloidal silica	Apply primer for 20 seconds; gently air blow; apply bond; gently air thin; light cure for 10 seconds

Bis-GMA: bisphenol-glycidyl methacrylate; HEMA: hydroxyethylmethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; CQ: di-camphorquinone.

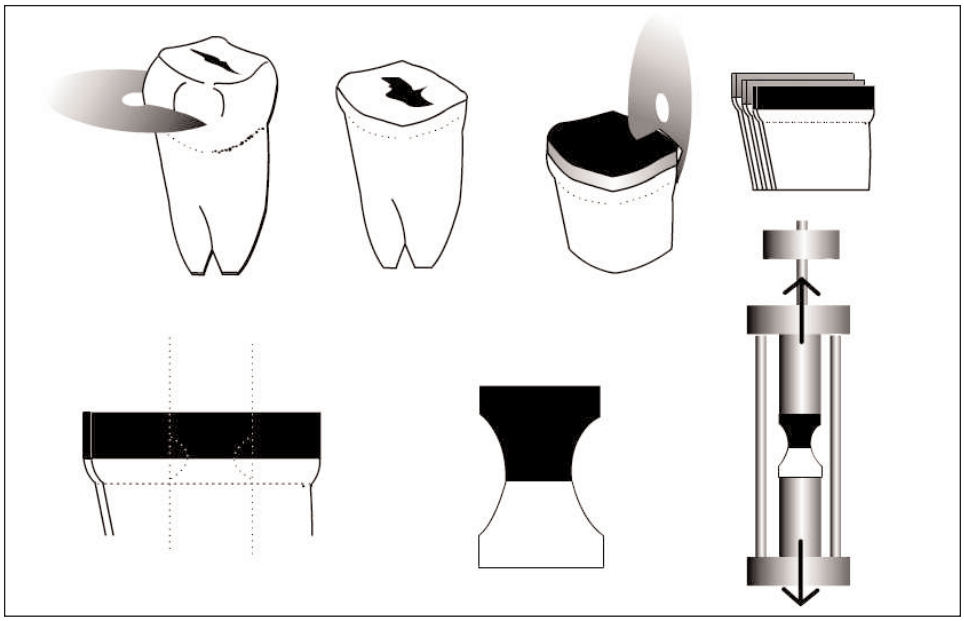


Figure 1. *Schematic diagram of the specimen preparation for the microtensile bond test; preparation of crown segment, preparation of a flat dentin surface, composite crown built up following bonding procedure, cutting slabs from bonded specimen, trimming into hour-glass shape and attachment to the testing device.*

Table 2: *Mean Microtensile Bond Strengths (MPa ± SD) of Resins to Sound vs Caries-affected Dentin*

Adhesive System	Sound Dentin	Caries-affected Dentin
Clearfil SE Bond	32.9 ± 13.7[a] (11)	15.9 ± 7.0[b] (10)
Clearfil SE Bond plus acid etching	19.2 ± 5.8[b] (12)	16.3 ± 5.7[b] (10)

Numbers in parentheses are the numbers of specimens tested. Groups with the same superscript number are not statistically significantly different (p>0.05).

RESULTS

Table 2 lists the mean microtensile bond strengths and standard deviations. The two-way ANOVA analysis revealed that there was a statistically significant interaction (p=0.012) between the type of dentin and the additional use of acid etching. The bond strengths of the Clearfil SE Bond system to sound dentin were significantly greater than to caries-affected dentin (p=0.000). However, there were no significant differences between sound and caries-affected dentin in bond strengths of the Clearfil SE Bond system with the additional use of acid etching (p=0.858).

According to the Tukey HSD multiple comparison test, phosphoric acid etching prior to the application of Clearfil SE Bond resulted in a significant decrease in bond strengths to sound dentin (p=0.003).

For caries-affected dentin, there were no statistically significant differences between the bond strengths developed by either group (p=1.000). Therefore, the use of additional acid etching had no positive effect on bond strength to caries-affected dentin.

DISCUSSION

In this study, the microtensile bond strength test was used to measure the bond strength of Clearfil SE Bond to sound versus caries-affected dentin as it permits the bond strength testing of variable surfaces such as sclerotic and caries-affected dentin. The microtensile bond strength test enables more accurate measurements of tensile bond strength because the typical hourglass design of the specimens imposes the highest uniform stress during testing (Sano & others, 1994). On the other hand, many specimens can be prepared from one tooth, which allows for a better comparison. In addition, this method permits the investigation of interfacial bond strengths on areas smaller than 1 mm^2 (Pashley & others, 1999).

The bond strength of Clearfil SE Bond to caries-affected dentin was lower than to sound dentin in this study. Therefore, the authors must accept the first null hypothesis. This finding is in agreement with other studies that have compared bond strengths to sound and caries-affected dentin (Nakajima & others, 1999; 2000a; Yoshiyama & others, 2000; 2002). It has been reported that the extent of demineralization by acid etching of the intertubular dentin and resin-impregnated dentin layer is related to the degree of tubule closure (Harnirattisai & others, 1992). In caries-affected dentin, many dentin tubules are occluded by crystalline deposits. The demineralization is greater in the areas of open dentin tubules of sound dentin than in the area of occluded tubules. Therefore, resin penetration may be deeper in sound dentin than in caries-affected dentin. As a result, the lower bond strengths to caries-affected dentin may be due to the lack of resin penetration into the tubules to form resin tags.

Another reason for the low bond strength values of caries-affected dentin could relate to the pH (acidity) of the adhesive system used. Self-etching adhesives can be divided into two subgroups, "mild" and "strong," depending on their pH and thus etching potential. In this study, the authors used the "mild" two-step self-etching adhesive Clearfil SE Bond (pH=2). The bonding mechanism of "mild" self-etching adhesives is based on hybridization, the difference being that only submicron hybrid layers are formed and resin-tag formation is less pronounced. In contrast to total-etch adhesives, collagen fibrils are not completely deprived of hydroxyapatite. This residual hydroxyapatite may serve as a receptor for additional intermolecular interactions with specific carboxyl or phosphate groups of functional monomers. These monomers may be able to interact more intimately with hydroxyapatite-coated collagen that otherwise would have almost completely lost its hydroxyapatite coating with a rather aggressive total-etch technique (Van Meerbeek & others, 2001).

It can be speculated that the Clearfil SE primer may not be acidic enough for the optimum etching of caries-affected dentin. In carious lesions, larger, less soluble calcium phosphate crystals occur. Thus, stronger acidity may be required to solubilize the mineral phase of caries-affected dentin. This self-etching adhesive might be unable to penetrate into the mineral-occluded dentinal tubules of caries-affected dentin. Another reason for the low bond strength values of caries-affected dentin might be related to the smear layer. The smear layers in caries-affected dentin may be more resistant to the action of self-etching primers as they include acid-resistant crystals and extrinsic proteins that have permeated into the mineral phase during demineralization cycles (Yoshiyama & others, 2000). The presence of crystals on dentin surfaces has been reported to lower resin-dentin bond strengths, because the resins bond to crystals within the smear layer rather than to the underlying dentin (Pashley, Tao & Pashley, 1993). Moreover, the acidity of the primer could also be buffered by the mineral components of the smear layer (Itou & others, 1994).

In this study, the bond strengths to sound dentin were decreased by the use of additional acid etching prior to applying Clearfil SE Bond. The Clearfil SE Bond system's primer has mild acidity, and thus does not disrupt or weaken the collagen as much as the phosphoric acid, resulting in higher bond strengths. Excessive etching of the dentin may also decrease bond strength. With additional acid etching, the dentin may be demineralized to a depth that might be inaccessible to complete resin impregnation. Strong acids tend to dissolve the inorganic part of dentin more deeply. It has been reported that dissolution of the hydroxyapatite can reach as far as 10 to 12 μm into the superficial dentin and may prevent the primer and adhesive from reaching the bottom of the demineralized dentinal network (Van Meerbeek & others, 1993). This might have weakened the resin-dentin bond. On the other hand, using a separate conditioning step to remove the smear layer defeats the original purpose of self-etching primers. Following treatment with self-etching primers, dentin surfaces are not rinsed and, therefore, there is no loss of mass from the dentin. This is thought to minimize the shrinkage or collapse of the collagen fibril network, which is consistent with an *in vitro* study by Nakajima and others (2000b), who found lower bond strength values when the dentin surfaces were etched with 40% phosphoric acid prior to applying self-etching primers. In contrast, Gordan and others (1997) found no significant differences between the bond strengths of Liner Bond 2 to dentin with and without prior 35% phosphoric acid etching.

One of the unexpected results of this study was the lack of significant differences in bond strengths to caries-affected dentin with the additional use of phosphoric acid pretreatment. Nakajima and others (2000a) suggested using stronger acids to solubilize the

intratubular mineral deposits of the caries-affected dentin in order to obtain optimal resin infiltration. In their study, they found that adhesive infiltrated less into the caries-affected dentin mineralized with 10% phosphoric acid compared to dentin etched with 32% phosphoric acid, resulting in lower bond strengths. On the other hand, in the current study, the use of acid etching prior to the application of Clearfil SE Bond yielded no improvements in bond strength to caries-affected dentin. This finding is in accordance with the manufacturer's claim that acid etching prior to placing the self-etching adhesive system is unnecessary.

During the development of carious lesions, repeated cycles of demineralization and remineralization occur, and the hardness of caries-affected dentin is lower than sound dentin (Fusayama, 1979; Ogawa & others, 1983; Marshall & others, 2001). Carious intertubular dentin exhibits a higher degree of porosity than sound intertubular dentin due to mineral loss (Hamid & Hume, 1997; Yoshiyama & others, 2002). Therefore, the depth of demineralization by additional phosphoric acid might be greater in caries-affected dentin and resin monomers might not be able to penetrate this deeply demineralized dentinal subsurface to produce resin hybridization of dentin (Sakoolnamarka & others, 2002). This may have contributed to the lower bond strengths. As a result, the second hypothesis, improvement of the bond strengths to both caries-affected and sound dentin by the additional use of acid etching prior to applying self-etching adhesive (Clearfil SE Bond) should be rejected.

CONCLUSIONS

The results of this *in vitro* study indicated that the microtensile bond strength of the self-etching adhesive Clearfil SE Bond to sound dentin was greater than to caries-affected dentin. While the use of phosphoric acid prior to the application of self-etching adhesive did not cause any significant differences in bond strengths for caries-affected dentin, it lowered bond strengths to sound dentin.

(Received 12 February 2003)

References

Fusayama T (1979) Two layers of carious dentin: Diagnosis and treatment *Operative Dentistry* **4(2)** 63-70.

Gordan VV, Vargas MA, Cobb DS & Denehy GE (1997) Evaluation of adhesive systems using acidic primers *American Journal of Dentistry* **10(5)** 219-223.

Haller B (2000) Recent developments in dentin bonding *American Journal of Dentistry* **13(1)** 44-50.

Hamid A & Hume WR (1997) Diffusion of resin monomers through carious dentin *in vitro Endodontics Dental Traumatology* **13(1)** 1-5.

Harnirattisai C, Inokoshi S, Shimada Y & Hosoda H (1992) Interfacial morphology of an adhesive composite resin and etched caries-affected dentin *Operative Dentistry* **17(6)** 222-228.

Itou K, Torii Y, Suziki K, Makai H & Inoue K (1994) Adhesion of restorative resin to tooth-adhesion promoted by Liner Bond II *Journal of Adhesive Dentistry* **12** 174-181.

Marshall GW, Habelitz, S, Gallagher R, Balooch M, Balooch G & Marshall SJ (2001) Nanomechanical properties of hydrated carious human dentin *Journal of Dental Research* **80(8)** 1768-1771.

Miyasaka K & Nakabayashi N (1999) Combination of EDTA conditioner and phenyl-P/HEMA self-etching primer for bonding to dentin *Dental Materials* **15(3)** 153-157.

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.

Nakajima M, Ogata M, Okuda M, Tagami J, Sano H & Pashley DH (1999) Bonding to caries-affected dentin using self-etching primers *American Journal of Dentistry* **12(6)** 309-314.

Nakajima M, Sano H, Urabe I, Tagami J & Pashley DH (2000a) Bond strengths of single-bottle dentin adhesives to caries-affected dentin *Operative Dentistry* **25(1)** 2-10.

Nakajima M, Ogata M, Harada N, Tagami J & Pashley DH (2000b) Bond strengths of self-etching primer adhesives to *in vitro*-demineralized dentin following mineralizing treatment *Journal of Adhesive Dentistry* **2(1)** 29-38.

Ogawa K, Yamashita Y, Ichijo T & Fusayama T (1983) The ultrastructure and hardness of the transparent layer of human carious dentin *Journal of Dental Research* **62(1)** 7-10.

Pashley EL, Tao L & Pashley DH (1993) Effects of oxalate on dentin bonding *American Journal of Dentistry* **6(3)** 116-118.

Pashley DH & Carvalho RM (1997) Dentin permeability and dentine adhesion *Journal of Dentistry* **25(5)** 355-372.

Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, Fernandes CA & Tay F (1999) The microtensile bond test: A review *Journal of Adhesive Dentistry* **1(4)** 299-309.

Sakoolnamarka R, Burrow MF, Kubo S & Tyas MJ (2002) Morphological study of demineralized dentine after caries removal using two different methods *Australian Dental Journal* **47(2)** 116-122.

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.

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

Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesives and cements to promote preservation dentistry *Operative Dentistry* **Supplement 6** 119-144.

Watanabe I, Nakabayashi N & Pashley DH (1994) Bonding to ground dentin by a phenyl-P self-etching primer *Journal of Dental Research* **73(6)** 1212-1220.

Yoshiyama M, Urayama A, Kimochi T, Matsuo T & Pashley DH (2000) Comparison of conventional vs self-etching adhesive bonds to caries-affected dentin *Operative Dentistry* **25(3)** 163-169.

Yoshiyama M, Tay FR, Doi J, Nishitani Y, Yamada T, Itou K, Carvalho RM, Nakajima M & Pashley DH (2002) Bonding of self-etch and total-etch adhesives to carious dentin *Journal of Dental Research* **81(8)** 556-560.

©Operative Dentistry, 2004, **29-2,** 182-187

Post-gel Polymerization Contraction of "Low Shrinkage" Composite Restoratives

AUJ Yap • MS Soh

Clinical Relevance

The polymerization contraction of "low-shrinkage" composites and ormocers was significantly lower than for conventional mini-filled composites.

SUMMARY

This study compared the post-gel contraction of two "low-shrinkage" composites (InTen-S [IS], Ivoclar-Vivadent; Aelite LS [AL], BISCO Inc) and an ormocer (Admira [AM], Voco) to two conventional mini-filled composites (Renew [RN], BISCO; Z100 [ZO], 3M ESPE). A strain-monitoring device and test configuration were used to measure the linear polymerization shrinkage associated with the various composites (A2 shade) during and up to 60 minutes post light polymerization. Each specimen was irradiated for 40 seconds using a halogen curing light (Max, Dentsply-Caulk) with an intensity of 401 mW/cm². Five specimens were made for each composite. Data was analyzed using one-way ANOVA/Scheffe's post-hoc test at significance level 0.05. The linear percentage shrinkage immediately after light polymerization and at 60 minutes post light polymerization ranged from 0.10 ± 0.02 to 0.40 ± 0.02% and 0.22 ± 0.02 to 0.60 ± 0.05%, respectively. Post-gel shrinkage ranking of the materials was as follows: immediately after light polymerization – IS < AL < AM < ZO < RN and at 60 minutes post light polymerization – IS < AL = AM < ZO < RN. The shrinkage associated with IS, AL and AM was significantly lower than for ZO and RN immediately after light polymerization and at 1, 10, 30 and 60 minutes post light polymerization. The post-gel polymerization shrinkage of IS, AL and AM was significantly lower than conventional mini-filled composites.

*Adrian UJ Yap, BDS, MSc, FAMS, FADM, FRSH, associate professor, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

MS Soh, BSc (Hons), research scholar, Department of Restorative Dentistry, Faculty of Dentistry, National University of Singapore, Republic of Singapore

*Reprint request: 5 Lower Kent Ridge Road, Singapore 119074, Republic of Singapore; e-mail: rsdyapuj@nus.edu.sg

INTRODUCTION

Composite materials and adhesive techniques have become the foundation of modern dentistry. Despite technological advances, polymerization shrinkage of composites still remains a challenge and imposes limitations in their clinical use (Sakaguchi & others, 1991; Dietschi & Dietschi, 1996, Davidson & Feilzer, 1997; Yap & others, 2000b). Composites contract about 1 to 5 volume percent during the polymerization process

(Davidson & Feilzer, 1997). The total shrinkage can be divided into pre-gel and post-gel phases. During pre-gel polymerization, the composite is able to flow and stresses within the structure are relieved (Davidson & de Gee, 1984). After gelation, flow ceases and cannot compensate for shrinkage stresses. Post-gel polymerization thus results in significant stresses in the surrounding tooth structure and composite-tooth bond (Feilzer, de Gee & Davidson, 1987). These stresses may produce defects in the composite-tooth bond, leading to bond failure, microleakage, post-operative sensitivity and recurrent caries. Such shrinkage stresses could also cause deformation of the surrounding tooth structure if the composite-tooth bond is good (Sheth, Fuller & Jensen, 1988), predisposing the tooth to fracture.

The effects of post-gel polymerization shrinkage are minimized in several ways, including applying liners/bases, incremental placement of composites and allowing the composites to contract freely to the adhesive interface (Davidson, 1986; Kemp-Scholte & Davidson, 1990; Davidson & Feilzer, 1997). A more recent method of minimizing shrinkage stress is to allow for composite flow during setting by means of controlled polymerization. This can be done by pre-polymerization at low light intensity followed by final cure at high intensity (soft-start polymerization), applying short-pulses of light energy (pulse activation) or a combination of the two. While some studies have shown that these polymerization modes may result in lower shrinkage stresses (Sakaguchi & Berge, 1998; Dennison & others, 2000), others have found no significant difference in shrinkage when compared to continuous cure modes (Koran & Kurschner, 1998; Silikas, Eliades & Watts, 2000; Yap, Ng & Siow, 2001; Yap, Soh & Siow, 2002).

Polymerization shrinkage may also be compensated for by mechanisms of expansion such as water sorption. The hygroscopic expansion of resin composites has been reported in many studies (Feilzer, de Gee & Davidson, 1990; Bastioli, Romano & Migliaresi, 1990; Yap & others, 2000b; Yap & Wee, 2002). Although such expansion may lead to substantial relaxation of polymerization contraction stresses, the water sorption process is slow and comes too late to be of clinical benefit (Davidson & Feilzer, 1997). New stresses may actually be generated due to volume gradient from the outer surface to the bulk of the restoration arising from the initial volume gain of surfaces exposed to the oral cavity. Thus, the solution to the polymerization shrinkage problem is to develop "low-shrinkage" and ideally "non-shrinking" resins for use in composite restoratives.

This study compared the post-gel contraction of two recently introduced "low-shrinkage" composites (InTen-S; Aelite LS) and an ormocer (Admira) to two conventional mini-filled composites (Renew; Z100) during and post light polymerization.

METHODS AND MATERIALS

Table 1 lists the composite materials evaluated and their manufacturers. The experimental set-up for measuring post-gel polymerization shrinkage/contraction was based on that used by Yap and others, 2000b; 2001; 2002. A diagrammatic representation of the test configuration for measuring polymerization shrinkage is shown in Figure 1. A glass slide served as the base of the set-up and a stiff black delrin frame (inner length 7.0 mm, width 4.0 mm and height 2.0 mm) was used to circumscribe the composite sample with the exception of a window for the strain gauge leads. Foil electrical resistance strain gauges (Foil Strain Gauge, RS Components Ltd, Singapore) were attached to flat surfaces on the glass slides. The gauges were 2 mm in length and had an electrical resistance 120 Ω and gauge factor 2.00. With the strain gauges in place, the different resin composites were placed into the cavity of the delrin frame. Care was taken to ensure complete filling of the frame, and excess composite material was extruded using pressure applied through a second glass slide and removed. The surface tack of the composite was adequate to ensure adhesion between the strain

Table 1: *The Composite Materials Evaluated*

Material	Manufacturer	Shade & Lot #	Composition
InTen-S	Ivoclar Vivadent, Schaan, Liechtenstein	A2 D51682	*Resins:* BisGMA, urethane dimethacrylate and BisEMA *Fillers:* Barium glass, ytterbium trifluoride *Filler volume:* 51%
Aelite LS	BISCO Inc, Schaumburg, IL, USA	A2 0200010647	*Resins:* Proprietary *Fillers:* Proprietary *Filler volume:* 74%
Admira	Voco, Cuxhaven, Germany	A2 18606	*Resins:* Inorganic-organic co-polymers, dimethacrylate *Fillers:* Inorganic glass *Filler volume:* 56%
Renew	BISCO Inc, Schaumburg, IL, USA	A2 0200010641	*Resins:* BisEMA, dimethacrylate *Fillers:* Barium glass, silica, titanium dioxide *Filler volume:* 59%
Z100	3M-ESPE, St Paul, MN, USA	A2 20020418	*Resins:* BisGMA, TEGDMA *Fillers:* Zirconia, silica *Filler volume:* 66%

BisGMA = Bisphenol-A-glycidyl methacrylate
BisEMA = ethoxylated BisGMA
TEGDMA = Triethylene glycol dimethacrylate

gauge and the composite materials. The leads from the strain gauge were connected to a strain monitoring device (Strain Gauge Recorder, Cole Parmer Instruments, IL, USA) initially balanced at zero. The strain monitoring device consisted of a chart recorder that functions by rationing sense voltage to signal voltage and converting it to analog output. Dimensional changes are thus effectively transferred to the gauges and measured in terms of resistance. Five specimens were made for each composite material.

The composite specimens were subsequently light polymerized using a conventional halogen light curing unit (Max; Dentsply-Caulk, Milford, DE, USA) with a mean output intensity of 401 ± 8 mW/cm^2 for 40 seconds. Dimensional change during and post light polymerization was monitored in air at room temperature (25 ± 1°C). Polymerization shrinkage measurements during light polymerization were taken at four-second intervals, while post light polymerization shrinkage measurements were taken at 0 (immediately after light polymerization), 1, 10, 30 and 60 minutes after removing the curing light. Percentage linear shrinkage was derived from the following equation: Percentage linear shrinkage ($\Delta L/L \times 100$) = ($\Delta R/R$)/K x 100, where ΔL = Change in length, L = Original length, ΔR = Change of resistance, R = Original resistance and K = Gauge factor (2). Data was subjected to one-way ANOVA and Scheffe's post-hoc tests at significance level 0.05.

RESULTS

Figure 2 shows the mean linear percent of shrinkage during light polymerization. Table 2 and Figure 3 show the mean linear percent of shrinkage immediately after removing the light source and at 1, 10, 30 and 60 minutes post light polymerization. The results of statistical analysis are reflected in Table 3.

At all time intervals, the least shrinkage was observed with InTen-S and the greatest with Renew. The ranking of post-gel shrinkage at 0, 1, 10 and 30 minutes post polymerization was the same and is as follows: InTen-S < Aelite LS < Admira < Z100 < Renew. Ranking at 60 minutes post polymerization was InTen-S < Aelite LS = Admira < Z100 < Renew. At all time intervals, post-gel shrinkage of InTen-S, Aelite LS and Admira were significantly lower than the mini-filled composites Z100 and Renew. At 1, 10 and 30 minutes post polymerization, the shrinkage of InTen-S was significantly lower than Admira. After 60 minutes post polymerization, both Aelite LS and Admira experienced significantly more shrinkage than InTen-S.

DISCUSSION

Light intensity and exposure duration have been shown to affect the cure of resin composites

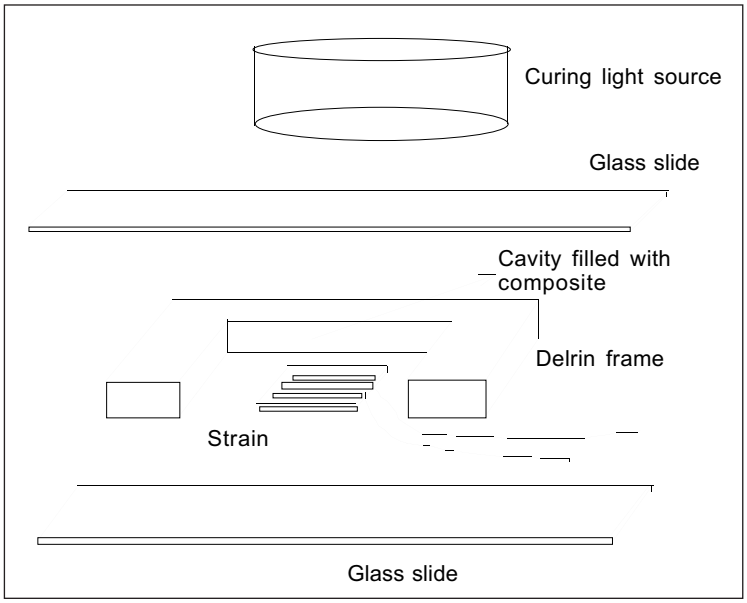


Figure 1. *Diagrammatic representation of the experimental set-up for the assessment of polymerization shrinkage.*

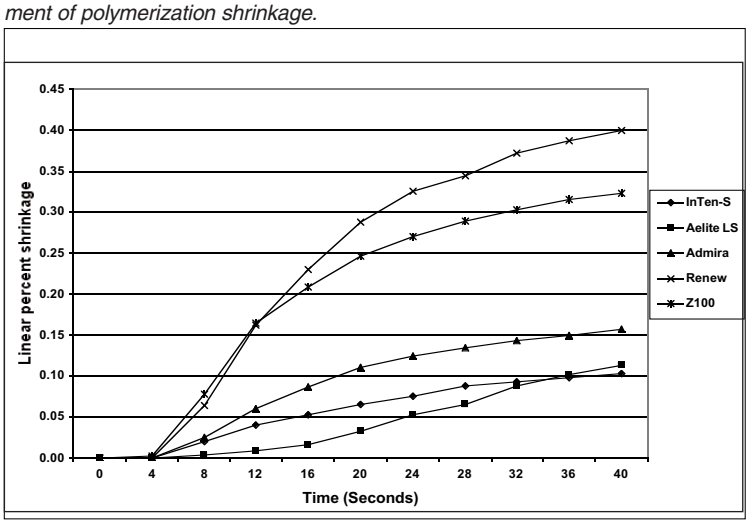


Figure 2. *Mean shrinkage during light polymerization.*

Table 2: *Mean Linear Percent Polymerization Shrinkage at the Various Post Light Polymerization Time Intervals*

Materials	0 Minute	1 Minute	10 Minutes	30 Minutes	60 Minutes
InTen-S	0.10 (0.02)	0.16 (0.02)	0.19 (0.02)	0.21 (0.02)	0.22 (0.02)
Aelite LS	0.11 (0.02)	0.19 (0.02)	0.25 (0.02)	0.29 (0.03)	0.31 (0.03)
Admira	0.16 (0.01)	0.23 (0.02)	0.28 (0.02)	0.30 (0.03)	0.31 (0.03)
Renew	0.40 (0.02)	0.50 (0.04)	0.55 (0.04)	0.58 (0.05)	0.60 (0.05)
Z100	0.32 (0.04)	0.38 (0.04)	0.41 (0.05)	0.43 (0.04)	0.45 (0.05)

Standard deviations in parentheses.

(Rueggeberg, Caughman & Curtis, 1994; Yap & Seneviratne, 2001). The light energy density (intensity x time) was kept constant for all materials as composite cure or polymerization that involves the conversion of monomer molecules into polymeric networks is accompanied by shrinkage. A curing unit with an intensity of approximately 400mW/cm^2 was used, as the latter is the minimum intensity recommended for routine polymerization of light-activated dental composites (Rueggeberg & others, 1994). Using higher intensity lights may result in increased shrinkage stresses (Feilzer & others, 1995; Venhoven, de Gee & Davidson, 1996). Since all curing light related factors, including curing tip distance, were standardized, any difference in polymerization shrinkage can be attributed to material factors. For all materials, the rate of shrinkage was greatest during the light polymerization reaction and continued after removal of the light source (Figure 2). A recent study (Yap & others, 2000b) reported that the post-polymerization dimensional changes of composite restoratives stabilized only after three days. A 60 minute evaluation was selected as shrinkage was found to be the greatest at this post-polymerization time period (Yap & others, 2000b).

Table 3: *Results of Statistical Analysis*

Time	Differences
0 minute	InTen-S, Aelite LS, Admira < Z100 < Renew
1 minute	InTen-S, Aelite LS, Admira < Z100 < Renew InTen-S < Admira
10 minutes	InTen-S, Aelite LS, Admira < Z100 < Renew InTen-S < Admira
30 minutes	InTen-S, Aelite LS, Admira < Z100 <Renew InTen-S < Admira
60 minutes	InTen-S < Aelite LS, Admira < Z100 < Renew

< denotes statistically significant differences. Results of one-way ANOVA/Scheffe's post-hoc test (p<0.05).

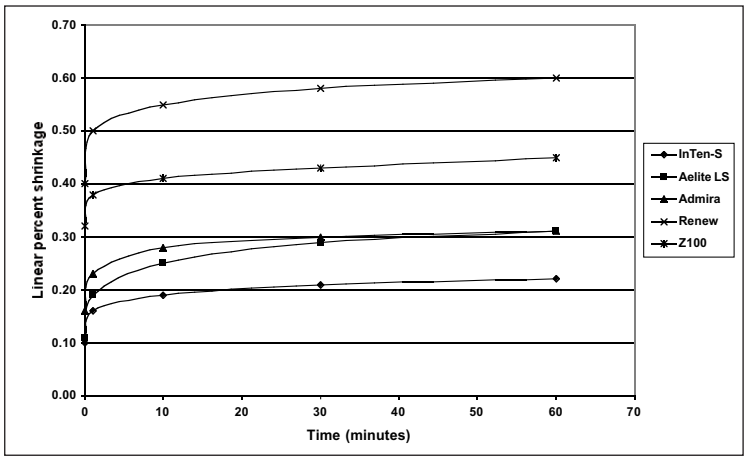


Figure 3. *Mean shrinkage post light polymerization.*

The primary material factors influencing polymerization shrinkage are the volume fraction of polymer matrix in the composite and the type of polymers used (Ferracane, 1995; O'Brien, 1997). Shrinkage is caused by the monomers becoming covalently bonded by the polymerization reaction, thus exchanging van der Waals' distances for covalent bond distances. The magnitude of shrinkage is dictated by the number of covalent bonds that form (polymer volume and extent of reaction) and the size of the monomers. Contraction per given volume of material can be minimized by using monomers with very large molecular weight (Ferracane, 1995). Polymerization shrinkage may not be a direct function of filler loading, as composites with high filler loads require low molecular weight monomers to ensure a proper handling viscosity (Davidson & Feilzer, 1997). Adding diluent monomers with low molecular weight increases polymerization shrinkage. The latter limits the amount of low molecular weight monomers that can be used in dental composites (Anusavice, 1996).

Eighty to 90% of commercial dental composites utilize Bisphenol-A-glycidyl methacrylate (BisGMA) monomer as their matrix-forming resin (Ruyter & Øysæd, 1987). Other base monomers used in commercial composites include urethane dimethacrylate (UDMA), urethane tetramethacrylate (UTMA), bis(methacryloyloxymethyl) tricyclodecane and ethoxylated bisphenol-A-dimethacrylate (BisEMA). BisGMA has a very high viscosity because of the hydrogen bonding interactions that occur between the hydroxyl groups on the monomer molecules. As a consequence, BisGMA must be diluted with a more fluid resin in order to be useful for dental composites (Ferracane, 1995). Triethylene glycol dimethacrylate (TEGDMA) has excellent viscosity and copolymerization characteristics and is often used as the diluent monomer for BisGMA-based composites. However, TEGDMA has been replaced with UDMA and BisEMA in several products to reduce shrinkage, aging and environmental effects (Yap, Low & Ong, 2000a). Both these resins have higher molecular weights than TEGDMA and therefore fewer double bonds per unit of weight. The aforementioned may account for the significantly lower shrinkage of InTen-S in spite of its relatively higher polymer content (49% volume). At 60 minutes post light polymerization, the linear percent shrinkage of InTen-S was significantly lower than all the other composites evaluated. These results corroborated the findings of a recent study where the polymerization shrinkage of InTen-S was found to be significantly lower than two other minifilled composites regardless of the intensity of the curing light (Yap, Wong & Siow, 2003).

The polymerization shrinkage of Aelite LS and Admira was significantly lower than Renew and Z100. At 60 minutes post light polymerization, the shrinkage

of Aelite LS and Admira was 30% to 50% lower than the mini-filled composites. According to the manufacturer, Aelite LS was designed to allow higher filler loading while maintaining a low viscosity and thus good handling. The filler loading of Aelite LS (74% volume) is higher than the other composites evaluated (51% to 66% volume). The significantly lower shrinkage of Aelite LS can be partially attributed to its low polymer volume (26% volume) and the proprietary resins used. The latter could not be disclosed as the patent for the resin formulation is still pending. The technology employed to reduce shrinkage of Admira is somewhat different. While composites are based on a purely organic resin matrix, the ormocer (organically modified ceramics) Admira consists of an inorganic-organic (inorganic backbone based on SiO_2 functionalized with polymerizable organic units) network matrix formed through polycondensation. These long ormocer molecule chains are very rigid and have low shrinkage during polymerization. Filler particles are embedded into the cross-linked inorganic-organic network matrix. The polymerization shrinkage of Renew was significantly greater than Z100 at all post-polymerization time intervals. This may be accounted for by its higher polymer volume (Renew - 41%; Z100 - 34%).

This study supports manufacturers' claims that InTen-S, Aelite LS and Admira had lower polymerization shrinkage than conventional composites. The use of these materials may reduce clinical problems associated with polymerization shrinkage of resin composites. However, the shrinkage associated with these products still ranges from 0.22% to 0.31%. As highlighted earlier, the ultimate solution to the polymerization shrinkage problem is the development of "non-shrinking" resins. Although earlier efforts to synthesize such resins were not successful, several developments in the last decade are more encouraging. Stansbury (1992) has synthesized spiro-orthocarbonate monomers (SOCs) that expand during polymerization through a double-ring opening process. Miyazaki and others (1994) reported on the development of acrylates and methacrylates containing spiro ortho esthers that were capable of being polymerized by heat, ionic and free radical initiators. The synthesis of new SOCs polymerized epoxy via cationic UV photo-initiation has also been reported (Byerley & others, 1992; Eick & others, 1993). Although these polymers are promising, problems balancing mechanical properties, water sorption, solubility and expansion still exist. The development of a dental composite with zero net polymerization dimensional change would be the single most significant advancement in these materials since their introduction in the late 1970s.

CONCLUSIONS

Under the conditions of this *in-vitro* study:

1. The rate of shrinkage of all composites was greatest during the light polymerization reaction and continued after removing the light source.
2. Ranking of shrinkage at the various post-polymerization time intervals was as follows: InTen-S < Aelite LS ≤ Admira < Z100 < Renew.
3. The polymerization shrinkage of "low-shrink" composites (InTen-S and Aelite LS) and ormocer (Admira) was significantly lower than conventional mini-filled composites (Z100 and Renew).

(Received 20 February 2003)

References

Anusavice KJ (1996) Restorative resins in *Phillips' Science of Dental Materials* 10th edition Philadelphia WB Saunders Co pp 273-200.

Bastioli C, Romano G & Migliaresi C (1990) Water sorption and mechanical properties of dental composites *Biomaterials* **11(3)** 219-223.

Byerley TJ, Eick JD, Chen GP, Chappelow CC & Millich F (1992) Synthesis and polymerization of new expanding dental monomers *Dental Materials* **8(6)** 345-350.

Davidson CL (1986) Resisting the curing contraction with adhesive composites *Journal of Prosthetic Dentistry* **55(4)** 446-447.

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 & Feilzer AJ (1997) Polymerization shrinkage and polymerization shrinkage stress in polymer-based restorations *Journal of Dentistry* **25(6)** 435-440.

Dennison JB, Yaman P, Seir R & Hamilton JC (2000) Effect of variable light intensity on composite shrinkage *Journal of Prosthetic Dentistry* **84(5)** 499-505.

Dietschi D & Dietschi JM (1996) Current developments in composite materials and techniques *Practical Periodontics and Aesthetic Dentistry* **8(7)** 603-613.

Eick JD, Byerley TJ, Chappell RP, Chen GP, Bowles CQ & Chappelow CC (1993) Properties of expanding SOC/epoxy copolymers for dental use in dental composites *Dental Materials* **9(2)** 123-127.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Feilzer AJ, de Gee AJ & Davidson CL (1990) Relaxation of polymerization contraction shear stress by hygroscopic expansion *Journal of Dental Research* **69(1)** 36-39.

Feilzer AJ, Dooren LH, de Gee AJ & Davidson CL (1995) Influence of light intensity on polymerization shrinkage and integrity of restoration-cavity interface *European Journal of Oral Science* **103(5)** 322-326.

Ferracane JL (1995) Current trends in dental composites *Critical Review in Oral Biology and Medicine* **6(4)** 302-318.

Kemp-Scholte CM & Davidson CL (1990) Complete marginal seal of Class V resin composite restorations effected by increased flexibility *Journal of Dental Research* **69(6)** 1240-1243.

Koran P & Kurschner R (1998) Effect of sequential versus continuous irradiation of a light-cured resin composite on shrinkage, viscosity, adhesion and degree of polymerization *American Journal of Dentistry* **11(1)** 17-22.

Miyazaki K, Takata T, Endo T & Inanaga A (1994) Thermal and photo-polymerization of (meth)acrylates containing a spiro ortho ester moiety and the properties of [(poly)methacrylate]s *Dental Materials* **13(1)** 9-18.

O'Brien WJ (1997) Polymeric restorative materials in *Dental Materials and Their Selection* 2nd edition Chicago Quintessence Publishing Co pp 97-114.

Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.

Ruyter IE & Øysæd H (1987) Composites for use in posterior teeth: Composition and conversion *Journal of Biomedical Materials Research* **21(1)** 11-23.

Sakaguchi RL & Berge HX (1998) Reduced light energy density decreases post-gel contraction while maintaining degree of conversion in composites *Journal of Dentistry* **26(8)** 695-700.

Sakaguchi RL, Sasik CT, Bunczak MA & Douglas WH (1991) Strain gauge method for measuring polymerization contraction of composite resoratives *Journal of Dentistry* **19(5)** 312-316.

Sheth JJ, Fuller JL & Jensen ME (1988) Cuspal deformation and fracture resistance of teeth with dentin adhesives and composites *Journal of Prosthetic Dentistry* **60(5)** 560-569.

Silikas N, Eliades G & Watts DC (2000) Light intensity effects on resin-composite degree of conversion and shrinkage strain *Dental Materials* **16(4)** 292-296.

Stansbury JW (1992) Synthesis and evaluation of new oxaspiro monomers for double ring-opening polymerization *Journal of Dental Research* **71(7)** 1408-1412.

Venhoven BA, de Gee AJ & Davidson CL (1996) Light initiation of dental resins: Dynamics of polymerization *Biomaterials* **17(24)** 2313-2318.

Yap AU, Low JS & Ong LF (2000a) Effect of food-simulating liquids on surface characteristics of composite and polyacid-modified composite restoratives *Operative Dentistry* **25(3)** 170-176.

Yap AU, Wang HB, Siow KS & Gan LM (2000b) Polymerization shrinkage of visible-light-cured composites *Operative Dentistry* **25(2)** 98-103.

Yap AU, Ng SC & Siow KS (2001) Soft-start polymerization: Influence on effectiveness of cure and post-gel shrinkage *Operative Dentistry* **26(3)** 260-266.

Yap AU & Seneviratne C (2001) Influence of light energy density on effectiveness of composite cure *Operative Dentistry* **26(5)** 460-466.

Yap AU, Soh MS & Siow KS (2002) Post-gel shrinkage with pulse activation and soft-start polymerization *Operative Dentistry* **27(1)** 81-87.

Yap AU & Wee KE (2002) Effects of cyclic temperature changes on water sorption and solubility of composite restoratives *Operative Dentistry* **27(2)** 147-153.

Yap AU, Wong NY & Siow KS (2003) Composite cure and shrinkage associated with very high intensity curing light *Operative Dentistry* **28(4)** 371-377.

©Operative Dentistry, 2004, **29-2,** 188-195

Influence of Different Bleaching Systems on Fracture Toughness and Hardness of Enamel

T Attin • T Müller
A Patyk • ÁM Lennon

Clinical Relevance

The use of external bleaching regimes may lead to the reduced fracture toughness of enamel.

SUMMARY

This study evaluated the influence of different bleaching procedures on the fracture toughness and microhardness of enamel. The labial aspects of 72 bovine incisors were prepared for microhardness determination. At baseline, Knoop hardness (KH) determination was conducted on each specimen. Moreover, the fracture toughness (FT) of enamel was assessed using Vickers hardness indentations with a load of 9.8 N. The length of both indentations and enamel cracks were recorded and used for calculation of FT. The samples were divided among six (A-F) groups (n=12) and sectioned, resulting in a control and an experimental half. The samples were stored in artificial saliva for 10 days. The experimental halves were removed from the saliva and subjected to bleaching according to manufacturers' instructions (A: Opalescence Xtra, B: Opalescence Quick, C: Rapid White, D: Whitestrips, E: Opalescence 10%, F: Opalescence PF 15%). Bleaching with C-F was conducted daily (C: twice per day for 10 minutes, D: twice per day for 30 minutes, E: 8 hours, F: 4 hours), systems A-B were applied on the first and fifth day (A: twice for 10 minutes, B: 1 hour). Finally, Knoop hardness and FT were assessed and statistically compared to baseline values using Wilcoxon-tests ($p<0.05$). KH and FT of the controls remained stable during storage in saliva. All bleaching regimens resulted in a statistically significant percentage loss of KH (mean + standard error of means): A: 17.3±2.8%, B: 8.6±3.3%; C: 83.5±0.61%, D: 29.0±1.9%, E: 9.0±2.9%, F: 5.4±2.2%. The percentage changes (mean + standard error of means) of FT in the experimental specimens were as follows: A: 3.9±9.5%, B: 0.1±4.7%; D: -8.2±7.1%, E: -18.9±4.7%, F: -12.0±4.7%. Due to severe surface softening, FT could not be determined for the samples in Group C. Applying Opalescence 10% resulted in a significant reduction in FT compared to baseline. In the remaining groups, changes in FT were not statistically significant.

*Thomas Attin, professor dr med dent, head of Department of Operative Dentistry, Preventive Dentistry and Periodontology, Georg-August-University Göttingen, Germany

Thomas Müller, dentist, private practice, Göttingen, Germany

Alfred Patyk, professor dr med dent, senior lecturer, Department of Prosthetic Dentistry, Georg-August-University Göttingen, Germany

Áine M Lennon, dr med dent, assistant professor, Department of Operative Dentistry, Preventive Dentistry and Periodontology, Georg-August-University Göttingen, Germany

*Reprint request: Robert-Koch-Str 40, D-37075 Göttingen, Germany; e-mail: thomas.attin@med.uni-goettingen.de

Bleaching with the tested materials resulted in 1) reduction of surface microhardness and 2) a decrease in fracture toughness (depending on the bleaching system applied).

INTRODUCTION

Vital tooth bleaching is becoming increasingly popular, showing good clinical long-term results and great patient satisfaction (Leonard, 1998). However, some studies demonstrate alterations to the histological aspects and composition of bleached enamel (Wandera & others, 1994). It has been shown that bleaching with 10% carbamide peroxide (CP) may result in a change in the calcium, phosphate and fluoride content in enamel (Crews & others, 1997; Perdigão & others, 1998; Potocnik, Kosec & Gaspersik, 2000; McCracken & Haywood, 1996; Burgmaier, Schulze & Attin, 2002). To date, macroscopically or clinically visible damage due to vital bleaching has not been described in the literature. Some authors, however, have reported slight surface alterations as assessed by scanning electron microscopy and a decrease in surface microhardness (Ernst, Marroquin & Willershausen-Zönnchen, 1996; Josey & others, 1996; Attin & others, 1997a; Attin & others, 2003); however, others have not found differences in microhardness. Changes in surface texture after applying carbamide peroxide bleaching gels are described as topographical alterations, decalcification and porosities in enamel (McGuckin, Babin & Meyer, 1992; Bitter & Sanders, 1993; Shannon & others, 1993). These slight alterations to the enamel surface did not become more severe *in vivo* as shown in a six month follow-up scanning electron microscopic study (Leonard & others, 2001). The loss of enamel microhardness due to carbamide peroxide gels is not limited to the enamel surface only but has also been detected in the subsurface within the outermost enamel layer (Cimilli & Pameijer, 2001; Akal & others, 2001; Basting, Rodrigues & Serra, 2001). Hardness changes observed after vital bleaching may depend on the applied product and its acidity (Rodrigues & others, 2001; Akal & others, 2001). With the bleaching gel's increasing acidity, alterations to the enamel structure are more likely to be observed (Shannon & others, 1993). In contrast to the above findings, there are also numerous investigations which found no signs of destruction or hardness loss of the bleached enamel surface compared to unbleached enamel (Haywood & others, 1990; Murchison, Charlton & Moore, 1992; Gultz & others, 1999; White & others, 2002).

A study by Seghi and Denry (1992) demonstrated a significant loss in enamel fracture toughness after bleaching with 10% carbamide peroxide. In their study, enamel samples were treated once with a 10% carbamide peroxide gel for 12 hours. The samples were not immersed in saliva after the bleaching procedure and fracture toughness was determined immediately after bleaching. That experimental design did not mimic the everyday situation very well. In the clinical situation, most bleaching therapies required more than one application. Moreover, both before and after bleaching, the teeth are in contact with saliva that remineralizes and rehardens bleached enamel for a period of time following the bleaching procedure when fluoride applications are performed (Attin & others, 1997a).

Consequently, the current study evaluated the influence of different bleaching procedures on the fracture toughness and surface microhardness of enamel in a model that simulates the interaction between saliva and bleached enamel *in vitro*.

METHODS AND MATERIALS

Seventy-two freshly extracted bovine incisors were stored in 1% thymol solution until used. The teeth were embedded in acrylic resin (Palavit G, Kulzer Wehrheim, Germany). The central aspect of the labial surface was ground flat and polished with water-cooled carborundum discs (4000 grit; Water Proof Silicon Carbide Paper Struers, Erkrath, Germany), thereby, removing approximately 200 µm of the outermost enamel layer. The thickness of the enamel that was removed was controlled with a micrometer (Mitutoyo, Tokyo, Japan). The bottom of the embedded specimens was aligned parallel to the polished surface with a thermoplastic material applied to the bottom of the samples (Figure 1).

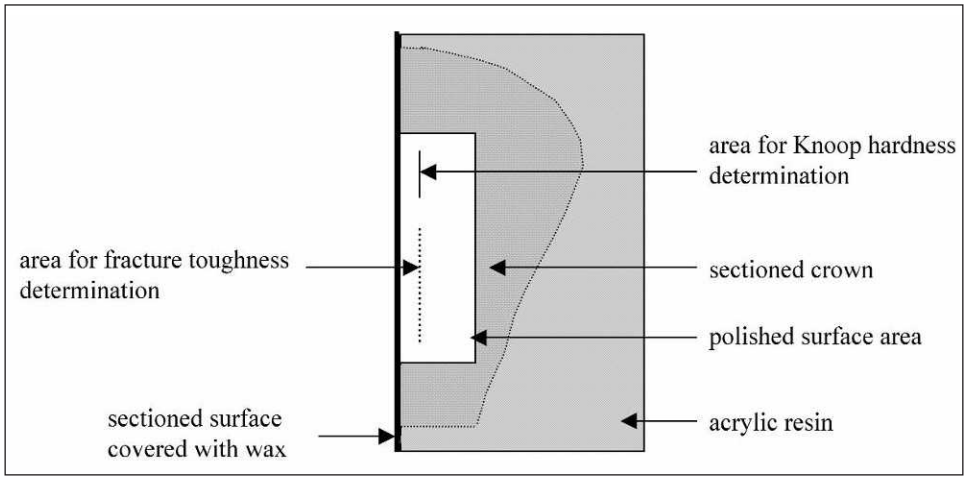


Figure 1. *Schematic drawing of an embedded, sectioned specimen.*

Table 1: *Compositions of the Tested Bleaching Products According to Material Safety Data Sheet Provided by the Respective Manufacturer; pH-values Were Determined as Described in the Text*

Product	Active Bleaching Substance	Composition, pH-value	Batch/Lot #
Opalescence Xtra	35% Peroxide	no information, pH=5.5	lot 47RY
Opalescence Quick	35% carbamide peroxide	no information, pH=7.0	lot 4DFQ
Rapid White	no information	accelerator: sodium chlorine whitening gel: aqua, glycerine, carbomer 974P, aroma, sodium hydroxide, citric acid, methylparaben, pH=3.7	accelerator: 1059414R2 whitening gel: 1051414E9
WhiteStrips	5.3% peroxide	purified water, glycerine, hydrogen peroxide, carbopol 956, sodium hydroxide, sodium acid pyrophosphate, sodium stannate, pH=6.4	lot/Pln/64/0430
Opalescence 10%	10% carbamide peroxide	no information, pH=7.8	lot 4F7R
Opalescence PF 15%	15% carbamide peroxide	potassium nitrate, fluoride, pH=7.9	lot 43MC

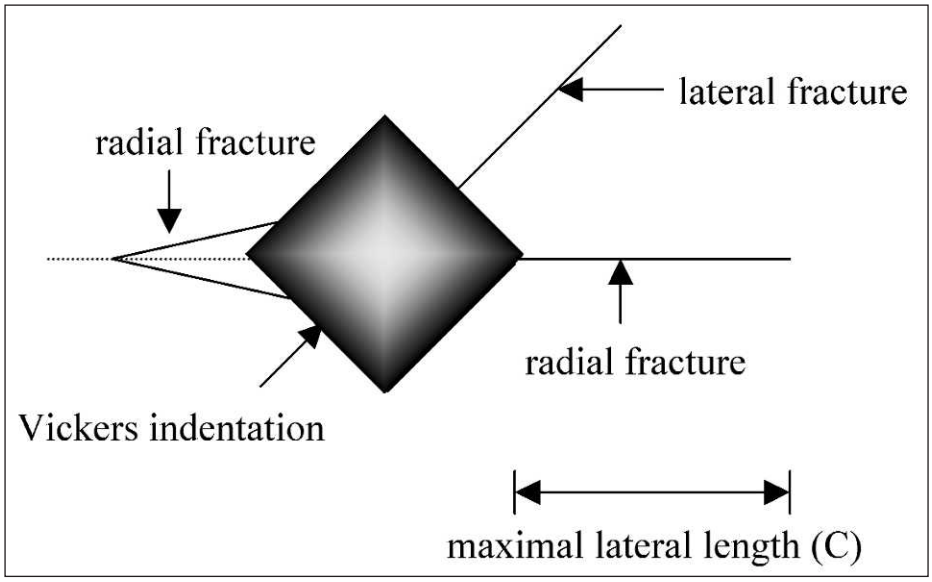


Figure 2. *Schematic drawing of a Vickers indentation with crack formation in enamel. The maximal length of the radial fracture was taken for calculation of fracture toughness.*

Prior to the experiment, all specimens were stored for 10 days at 37°C in artificial saliva (Attin & others, 2000) that was renewed every day. The artificial saliva (pH 6.4) contains, among other things, 1.53 mmol of calcium and 4.82 mmol of inorganic phosphate. Every 12 specimens were stored in 300 ml of saliva.

After 10-day storage, baseline Knoop microhardness was determined. The specimens were then evenly distributed among six experimental groups (A-F) according to their Knoop microhardness values. Stratified random sampling was applied so that the average microhardness in the six groups was nearly equal. Moreover, fracture toughness of the enamel was assessed performing Vickers hardness indentations as described below. Knoop hardness and fracture toughness were performed in the central part of the exposed enamel surface. The specimens were then sectioned along their central line with a water-cooled saw (Exakt Apparatebau, Norderstedt, Germany), resulting in a control and an experimental half. The experimental and control halves were covered with wax, so that only the flattened enamel surfaces of the samples were exposed to the bleaching regimes.

Experiment

The samples were again transferred to artificial saliva that was replaced with fresh saliva daily for 10 days. The experimental halves were removed from the saliva and subjected to bleaching according to manufacturers' instructions as described below [A: Opalescence Xtra (35% H_2O_2), B: Opalescence Quick (35% CP), C: Rapid White (peroxide free), D: Whitestrips (5.3% H_2O_2), E: Opalescence (10% CP), F: Opalescence PF (15% CP]. Bleaching with C-F was conducted daily and systems A-B were applied on the first and fifth day.

Prior to bleaching, the enamel surfaces were dried with cotton pellets. After bleaching and before re-transferring to saliva, the bleaching substances were carefully removed with a soft toothbrush under running tap water.

Group A (O-Xtra: Opalescence Xtra, Ultradent Products, South Jordan, UT, USA): The samples were covered at the first and fifth day with a 1-mm thick layer of Opalescence Xtra for 10 minutes at room temperature. After three and six minutes, respectively, the bleaching agent was activated with a halogen polymerization light (Elipar-Classic, ESPE, Seefeld, Germany)

for 10 seconds. A specially designed gadget guaranteed that the distance between the tip of the polymerization device and the enamel surface constantly remained at 8 mm.

Group B (O-Quick: Opalescence Quick, Ultradent Products): The specimens were covered with a 1-mm thick layer of Opalescence Quick for one hour on the first and fifth day. Bleaching was performed in a humid atmosphere at 37°C.

Group C (Rapid: Rapid White, Rapid White Products, Tonawanda, NY, USA): Rapid White accelerator was applied to the enamel surfaces followed by coverage with a 1-mm thick layer of Rapid White gel in a humid atmosphere at 37°C. Bleaching was conducted twice daily for 10 minutes with eight hours elapsing between the two applications.

Group D (White: Whitestrips, Procter & Gamble, Cincinnati, OH, USA): The enamel samples were covered with a strip twice a day for 30 minutes (humid atmosphere at 37°C) with eight hours elapsing between the two applications.

Group E (O-10%: Opalescence 10%; Ultradent): On each of the 10 days, the samples were covered with a 1-mm layer of the bleaching gel for eight hours in a humid atmosphere at 37°C.

Group F (O-15%: Opalescence PF 15%; Ultradent): On each of the 10 days, the samples were covered with a 1-mm layer of the bleaching gel for four hours in a humid atmosphere at 37°C.

Approximately 0.5 g of the bleaching substances were dissolved in distilled water for pH-determination with a pH-meter (pH/ION Meter pmX 3000 WTW, Weilheim, Germany). Table 1 shows the PH-values and additional information about the bleaching agents.

Analysis

Surface microhardness and enamel fracture toughness were assessed directly after polishing the unsectioned specimens (baseline values) and after the 10-day bleaching period (experimental halves) and storage in artificial saliva (control halves), respectively. Microhardness was determined using a Knoop diamond (Mikrohärteprüfgerät, Durimeter, Ernst Leitz GmbH, Wetzlar, German) at a load of 0.25 N applied for 15 seconds. The flattened enamel surface was large enough that enough space (about 50 µm) was given for the indentations without interfering with each other. At each period in time, 10 indentations were performed on each half of the respective specimen and averaged.

Fracture toughness was assessed by applying Vickers hardness indentations with a load of 9.8 N hardness testing device (Typ 3212001, Zwick, Ulm, Germany). The Vickers hardness indentations were performed on areas of the enamel surface that were different from the Knoop hardness measurements (Figure 2). The space between the Vickers indentations amounted to a distance of 500 µm. The indentations were measured under a light microscope (40-fold magnification) with an X/Y-measuring table (Z-502, RSF–Elektronik, Tarsdorf, Austria). For evaluation of the enamel fracture toughness, the diagonal length of the Vickers hardness indentation was first measured and the hardness calculated according to the following formula given by Lawn and Marshall (1979):

$$\text{Vickers hardness (HV)} = 0.47 \quad \frac{P}{a^2}$$

with: P=applied load [MN] (here: $9{,}807 \times 10^{-6}$ MN)

a=half of the diagonal length of indentation [m]

The length of the maximal radial fracture line was then determined (Figure 2) and used to calculate the fracture toughness according to Anstis and others (1981):

$$\text{Fracture toughness (K)} = 0.016 \times \left(\frac{E}{H}\right)^{1/2} \times \frac{P}{c^{3/2}}$$

with: H=Vickers hardness (HV) [GPa]

c=maximal lateral length of fracture line [m]

P=applied load [MN]

E=Young modulus [GPa]

Young modulus for human enamel is 84.1 GPa (Craig, Peyton & Johnson, 1961). Since the composition of bovine enamel is similar to human enamel (Esser, Tinschert & Marx, 1998), the authors chose 84.1 GPa for the constant value E of the formula.

Statistical Analysis

Knoop hardness and fracture toughness of the experimental and control halves were statistically compared to the baseline values using Wilcoxon-tests ($p<0.05$). Statistical analysis was performed with the software package Statistica 6.0 (Statsoft, Tulsa, OK, USA). Moreover, fracture toughness of each specimen after the 10-day period was calculated as percentage change with respect to baseline toughness of the specimen.

Since the tested bleaching systems were very different with respect to application procedure, no direct statistical comparisons between the systems were applied.

RESULTS

Figure 3 depicts the mean Knoop microhardness values determined at baseline and in the controls and test samples at the end of the experiment. Statistical analysis revealed significant differences ($p<0.05$) between baseline values and the respective test specimens for all bleaching materials. No significant difference was recorded for hardness of the controls compared to the respective baseline values in any of the groups. Figure 4 shows the results of the Vickers hardness determina-

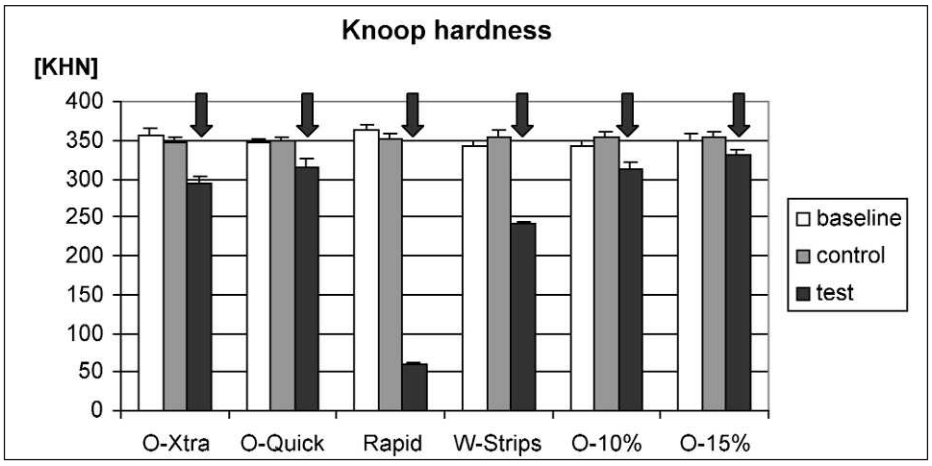


Figure 3. *Mean Knoop hardness values in specimens at baseline and after treatment. Statistically significantly different values are marked with arrows (vertical lines: standard error of means).*

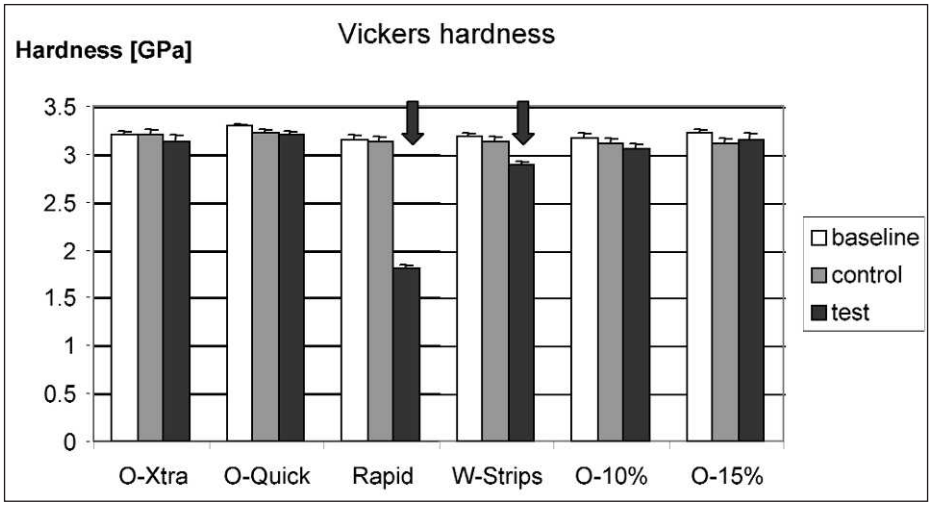


Figure 4. *Mean Vickers hardness values in specimens at baseline and after treatment. Statistically significantly different values are marked with arrows (vertical lines: standard error of means).*

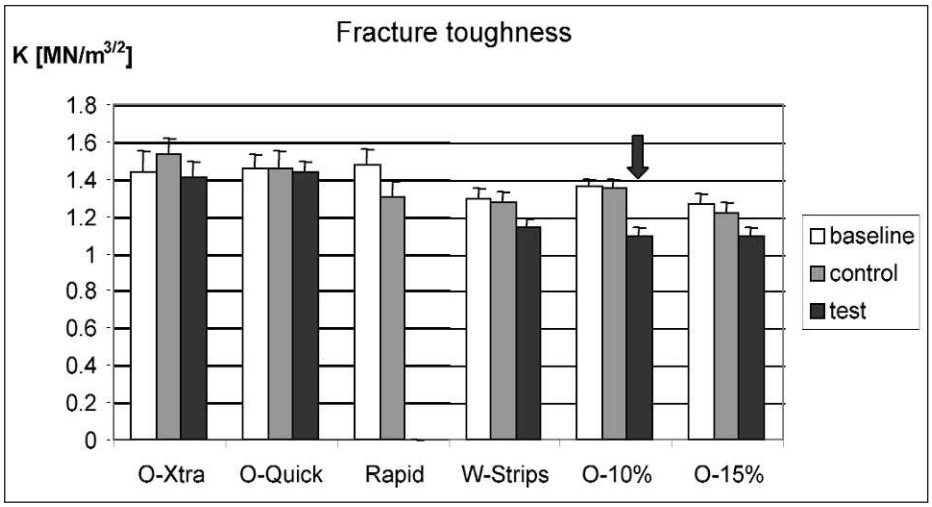


Figure 5. *Mean fracture toughness in specimens at baseline, and after treatment. Statistically significantly different values are marked with arrows (vertical lines: standard error of means).*

tion. Vickers hardness remained stable during the experiment in all groups (controls and test) except for the test samples treated with Rapid White and Whitestrips, respectively. In these two groups, a significant Vickers microhardness reduction was observed compared to baseline values (Rapid White: p=0.002; Whitestrips: p=0.005).

Figure 5 shows the mean values recorded for fracture toughness of the enamel samples. Fracture toughness in the samples treated with Rapid White could not be determined since the softened surface of those specimens did not allow for generating Vickers indentations showing crack formations. Only the test group treated with Opalescence 10% showed a significant reduction in fracture toughness at the end of the experiment (p=0.008). The remaining samples did not reveal any significant differences compared to the respective baseline values. This was true for all controls and the test groups.

Table 2 depicts the percentage changes of fracture toughness. Only minimal changes were recorded for Opalescence Xtra and Opalescence Quick. However, for Whitestrips, Opalescence 10% and Opalescence 15%, the reduction in enamel fracture toughness ranged between 8.2% and 18.9%.

DISCUSSION

In this study, a model comprising periods of treatment with bleaching agents and remineralization in artificial saliva was utilized, thereby, attempting to simulate physiological conditions during bleaching procedures. Further studies should prove how far intraoral conditions during remineralization of bleached dental hard tissue are reflected by the applied model. Application of the bleaching agents was performed as recommended by the manufacturers. The gels (Opalescence Quick, 10 and 15% CP), Rapid White and the Whitestrips were applied to the enamel samples in a humid atmos-

phere at 37° in an attempt to reflect the intraoral clinical situation during home-bleaching. Opalescence Xtra is designed for chairside bleaching under isolation of the teeth using a rubber dam. Therefore, Opalescence Xtra was applied to the enamel surfaces at room temperature without simulating the humid atmosphere of the oral cavity. Bovine teeth were used as a substitute for human teeth, as in other investigations (Ruse & others, 1990; Ten Cate & Arends, 1977; Attin & others, 1997a; Attin & others, 2003). Although the composition and physical properties of bovine enamel are very similar to human enamel, it should be noted that the flexural strength of bovine enamel is higher compared to human enamel (Esser & others, 1998). The use of bovine teeth instead of human teeth had the advantage that the flattened and polished surface areas needed for microhardness and fracture toughness measurements were large enough to allow for performing multiple microhardness indentations on the surface without mutual interference.

Table 2: *Mean (standard error of means), Minimum and Maximum Percentage Changes [%] of Fracture Toughness at the End of the Experiment in Enamel Specimens Bleached with Different Products as Related to Respective Baseline Values (n=12)*

Product	Mean	Minimum	Maximum
Opalescence Xtra	3.9 (9.5)	-38.5	75.6
Opalescence Quick	0.1 (4.7)	-20.6	35.2
Rapid White	-#	-	-
WhiteStrips	-8.2 (7.2)	-46.6	33.4
Opalescence 10%	-18.9 (4.7)	-42.1	15.3
Opalescence PF 15%	-12.0 (4.7)	-37.7	12.0

#In the specimens treated with Rapid White, fracture toughness could not be determined with the kind of measurement applied due to severe softening of the surface.

During the experiment, the enamel specimens were stored in artificial saliva that, based on several *in vitro* studies, has been proven to act as an effective agent for rehardening softened dental enamel (Attin, Zirkel & Hellwig, 1998; Attin, Deifuss & Hellwig, 1999; Attin & others, 2000; Klimek & Hellwig, 1989; Klimek, Hellwig & Ahrens, 1982).

In this investigation, surface alteration was assessed with microhardness measurements judged to be an appropriate tool to investigate the surface softening of enamel (Curzon & Hefferren, 2001). In this study, the enamel surfaces showed Knoop microhardness indentations from about 31 to 33 µm (Groups A, B, D, E and F) and 80 µm (Rapid White) in length, respectively. These indentation lengths are equivalent to penetration depths of the Knoop diamond of 1.02 to 1.08 µm (Rapid White 2.63 µm), so that the results of the Knoop microhardness determination in the current study predominantly reflect alterations of the superficial enamel layer.

Treatment with bleaching agents resulted in Knoop microhardness loss in all groups. This finding is in agreement with previous investigations that also demonstrated a decrease in microhardness of enamel due to bleaching (Josey & others, 1996; Cimilli & Pameijer, 2001; Akal & others, 2001; Basting & others, 2001; Burgmaier & others, 2002; Attin & others, 1997a; Attin & others, 2003). In this study, surface softening was very pronounced after applying Rapid White (Group C). Rapid White showed an acidic pH of 3.7 and contains citric acid among other things. Citric acid is known to demonstrate an erosive potential and softens the surfaces of dental hard tissue (Lussi, Jaeggi & Jaeggi-Schärer, 1995; Attin & others, 1997b). Surface softening of enamel, as observed as a sequela to erosive attacks with acidic substances, leads to increased susceptibility to surface loss due to abrasive influences such as toothbrushing (Attin & others, 1997b; Davis & Winter, 1980). Further studies must prove whether the reduction in surface hardness after external bleaching procedures has an influence on the susceptibility of enamel to abrasive wear also.

In this study, enamel fracture toughness was determined with an indentation technique described by Anstis and others (1981). Seghi and Denry (1992) also applied this method; they investigated the influence of bleaching on enamel fracture toughness. In addition, this technique is well established for fracture toughness measurements of (predominately) brittle dental restorative materials, such as ceramics (Morena, Lockwood & Fairhurst, 1986; Jones & others, 1988). In the study, fracture toughness measurement was not possible in the samples treated with Rapid White. Due to severe softening of the enamel surfaces in the specimens in this group, placement of the indentations did not result in the crack formation required for determining fracture toughness with the indentation technique.

In the above mentioned study by Seghi and Denry (1992), enamel fracture toughness was significantly reduced after a single eight-hour application of a 10% carbamide peroxide whitening gel. In the present study, fracture toughness was significantly reduced only in the samples treated with Opalescence 10% for eight hours daily. In the remaining groups with higher concentrations of the active bleaching substance (peroxide), shorter treatment periods were applied each day. In these groups, no (significant) reductions in fracture toughness were observed. A previous study had shown that the color change observed directly after completing the bleaching therapy with carbamide peroxide gels

was not completely stable during the post-bleaching period but stabilized to a constant level after six weeks (Matis & others, 1998). The slight reversal in color observed in the post-bleaching period in this study might have been caused by rehydration of the teeth, which supposedly became dehydrated during bleaching with a gel. It may be speculated that longer application times of low level carbamide peroxide gel in the current study resulted in more pronounced dehydration compared to the other bleaching regimes, thereby, leading to increased brittleness of the enamel with a decreased resistance against crack formation. In theoretical models, decreased fracture toughness of brittle materials claimed to lead to an increase in abrasive wear (Mair & others, 1996). In this model, it was suggested that the facilitated crack formation might result in greater volumes of material removed in case of abrasive influences. Further studies must prove whether reduced fracture toughness of the bleaching agent Opalescence 10% observed in this study correlates with an increased susceptibility to abrasion.

Determining microhardness and fracture toughness was performed for five days (Group A and B) and one day (Group C-F) after completing bleaching therapy, respectively. During this final period the samples were stored in artificial saliva. Microstructural alterations in bleached enamel may be reversed due to repair by salivary components (Attin & others, 1997a; de Freitas & others, 2002). It may be speculated that, in this study, the remineralizing periods after completing bleaching therapy were not long enough to repair the microstructural defects. Studies must evaluate how long it will take after bleaching to reverse deterioration of the physical properties of enamel observed in the study.

Interestingly, the loss of surface microhardness and fracture toughness was nearly similar for Opalescence 10% and Opalescence PF 15%. It should be noted that Opalescence PF 15% contains fluoride, which may have contributed to better remineralization after bleaching (Attin & others, 2003), thereby, leading to similar surface alterations as compared to the lower concentrated Opalescence 10%.

CONCLUSIONS

Due to the results of this investigation, it is concluded that bleaching with the tested materials results in 1) reduction of surface microhardness and 2) decreased fracture toughness (depending on the applied bleaching system).

Acknowledgements

The study was partly supported by a grant from Ultradent Products, South Jordan, UT, USA.

(Received 4 March 2003)

References

Akal N, Over H, Olmez A & Bodur H (2001) Effects of carbamide peroxide containing bleaching agents on the morphology and subsurface hardness of enamel *Journal of Clinical Pediatric Dentistry* **25(4)** 293-296.

Anstis GR, Chantikul P, Lawn BR & Marshall DB (1981) A critical evaluation of indentation techniques for measuring fracture toughness: I, Direct crack measurement *Journal of the American Ceramic Society* **64** 533-538.

Attin T, Buchalla W, Gollner M & Hellwig E (2000) Use of variable remineralization periods to improve the abrasion resistance of previously eroded enamel *Caries Research* **34(1)** 48-52.

Attin T, Deifuss H & Hellwig E (1999) Influence of acidified fluoride gel on abrasion resistance of eroded enamel *Caries Research* **33(2)** 135-139.

Attin T, Kielbassa AM, Schwanenberg M & Hellwig E (1997a) Effect of fluoride treatment on remineralization of bleached enamel *Journal of Oral Rehabilitation* **24(4)** 282-286.

Attin T, Kocabiyik M, Buchalla W, Hannig C & Becker K (2003) Susceptibility of enamel surfaces to demineralization after application of fluoridated carbamide peroxide gels *Caries Research* **37(2)** 93-99.

Attin T, Koidl U, Buchalla W, Schaller HG, Kielbassa AM & Hellwig E (1997b) Correlation of microhardness and wear of differently eroded bovine dental enamel *Archives of Oral Biology* **42(3)** 243-250.

Attin T, Zirkel C & Hellwig E (1998) Brushing abrasion of eroded dentin after application of sodium fluoride solutions *Caries Research* **32(5)** 344-350.

Basting RT, Rodrigues AL Jr & Serra MC (2001) The effect of 10% carbamide peroxide bleaching material on microhardness of sound and demineralized enamel and dentin *in situ Operative Dentistry* **26(6)** 531-539.

Bitter NC & Sanders JL (1993) The effect of four bleaching agents on the enamel surface: A scanning electron microscopic study *Quintessence International* **24(11)** 817-824.

Burgmaier GM, Schulze IM & Attin T (2002) Fluoride uptake and development of artificial erosions in bleached and fluoridated enamel *in vitro Journal of Oral Rehabilitation* **29(9)** 799-804.

Cimilli H & Pameijer CH (2001) Effect of carbamide peroxide bleaching agents on the physical properties and chemical composition of enamel *American Journal of Dentistry* **14(2)** 63-66.

Craig RG, Peyton FA & Johnson DW (1961) Compressive properties of enamel, dental cements, and gold *Journal of Dental Research* **40(5)** 936-945.

Crews KM, Duncan D, Lentz D, Gordy FM & Tolbert B (1997) Effect of bleaching agents on chemical composition of enamel *Mississippi Dental Association Journal* **53(1)** 20-21.

Curzon ME & Hefferren JJ (2001) Modern methods for assessing the cariogenic and erosive potential of foods *British Dental Journal* **191(1)** 41-46.

Davis WB & Winter PJ (1980) The effect of abrasion on enamel and dentine after exposure to dietary acid *British Dental Journal* **148(11-12)** 253-256.

de Freitas PM, Basting RT, Rodrigues JA & Serra MC (2002) Effects of two 10% peroxide carbamide bleaching agents on dentin microhardness at different time intervals *Quintessence International* **33(5)** 370-375.

Ernst CP, Marroquin BB & Willershausen-Zönnchen B (1996) Effects of hydrogen peroxide-containing bleaching agents on the morphology of human enamel *Quintessence International* **27(1)** 53-56.

Esser M, Tinschert J & Marx R (1998) [Materialkennwerte der Zahnhartsubstanz des Rindes im Vergleich zur humanen Zahnhartsubstanz] *Deutsche zahnärztliche Zeitschrift* **53(10)** 713-717.

Gultz J, Kaim J, Scherer W & Gupta H (1999) Two in-office bleaching systems: A scanning electron microscope study *Compendium of Continuing Education in Dentistry* **20(10)** 965-972.

Haywood VB, Leech T, Heymann HO, Crumpler D & Bruggers K (1990) Nightguard vital bleaching: Effects on enamel surface texture and diffusion *Quintessence International* **21(10)** 801-804.

Jones DW, Rizkalla AS, King HW & Sutow EJ (1988) Fracture toughness and dynamic modulus of tetrasilicic-micaglass-ceramic *Journal of the Canadian Ceramic Society* **57** 39-46.

Josey AL, Meyers IA, Romaniuk K & Symons AL (1996) The effect of a vital bleaching technique on enamel surface morphology and the bonding of composite resin to enamel *Journal of Oral Rehabilitation* **23(4)** 244-250.

Klimek J & Hellwig E (1989) [Beeinflussung der De- und Remineralisation von Zahnschmelz durch Zähneputzen mit einem Zahnsalz] *Oralprophylaxe* **11(1)** 26-30.

Klimek J, Hellwig E & Ahrens G (1982) Fluoride taken up by plaque, by the underlying enamel and by clean enamel from three fluoride compounds *in vitro Caries Research* **16(2)** 156-161.

Lawn BR & Marshall DB (1979) Hardness, toughness, and brittleness: An indentation analysis *Journal of the American Ceramic Society* **62** 347-350.

Leonard RH Jr (1998) Efficacy, longevity, side effects, and patient perceptions of nightguard vital bleaching *Compendium of Continuing Education in Dentistry* **19(8)** 766-781.

Leonard RH Jr, Eagle JC, Garland GE, Matthews KP, Rudd AL & Phillips C (2001) Nightguard vital bleaching and its effect on enamel surface morphology *Journal of Esthetic and Restorative Dentistry* **13(2)** 132-139.

Lussi A, Jaeggi T & Jaeggi-Schärer S (1995) Prediction of the erosive potential of some beverages *Caries Research* **29(5)** 349-354.

Mair LH, Stolarski TA, Vowles RW & Lloyd CH (1996) Wear: Mechanisms, manifestations and measurement. Report of a workshop *Journal of Dentistry* **24(1-2)** 141-148.

Matis BA, Cochran MA, Eckert G & Carlson TJ (1998) The efficacy and safety of a 10% carbamide peroxide bleaching gel *Quintessence International* **29(9)** 555-563.

McCracken MS & Haywood VB (1996) Demineralization effects of 10 percent carbamide peroxide *Journal of Dentistry* **24(6)** 395-398.

McGuckin RS, Babin JF & Meyer BJ (1992) Alterations in human enamel surface morphology following vital bleaching *Journal of Prosthetic Dentistry* **68(5)** 754-760.

Morena R, Lockwood PE & Fairhurst CW (1986) Fracture toughness of commercial dental porcelains *Dental Materials* **2(2)** 58-62.

Murchison DF, Charlton DG & Moore BK (1992) Carbamide peroxide bleaching: Effects on enamel surface hardness and bonding *Operative Dentistry* **17(5)** 181-185.

Perdigão J, Francci C, Swift EJ Jr, Ambrose WW & Lopes M (1998) Ultra-morphological study of the interaction of dental adhesives with carbamide peroxide-bleached enamel *American Journal of Dentistry* **11(6)** 291-301.

Potocnik I, Kosec L & Gaspersik D (2000) Effect of 10% carbamide peroxide bleaching gel on enamel microhardness, micostructure, and mineral content *Journal of Endodontics* **26(4)** 203-206.

Rodrigues JA, Basting RT, Serra MC & Rodrigues AL Jr (2001) Effects of 10% carbamide peroxide bleaching materials on enamel microhardness *American Journal of Dentistry* **14(2)** 67-71.

Ruse ND, Smith DC, Torneck CD & Titley KC (1990) Preliminary surface analysis of etched, bleached, and normal bovine enamel *Journal of Dental Research* **69(9)** 1610-1613.

Seghi RR & Denry I (1992) Effects of external bleaching on indentation and abrasion characteristics of human enamel *in vitro Journal of Dental Research* **71(6)** 1340-1344.

Shannon H, Spencer P, Gross K & Tira D (1993) Characterization of enamel exposed to 10% carbamide peroxide bleaching agents *Quintessence International* **24(1)** 39-44.

Ten Cate JM & Arends J (1977) Remineralization of artificial enamel lesions *in vitro Caries Research* **11(5)** 277-286.

Wandera A, Feigal RJ, Douglas WH & Pintado MR (1994) Home-use tooth bleaching agents: An *in vitro* study on quantitative effects on enamel, dentin and cementum *Quintessence International* **25(8)** 541-546.

White DJ, Kozak KM, Zoladz JR, Duschner H & Gotz H (2002) Peroxide interactions with hard tissues: Effects on surface hardness and surface/subsurface ultrastructural properties *Compendium of Continuing Education in Dentistry* **23(1A)** 42-48.

©Operative Dentistry, 2004, **29-2**, 196-202

Effective Bond Strength of Current Adhesive Systems on Deciduous and Permanent Dentin

P Senawongse • C Harnirattisai
Y Shimada • J Tagami

Clinical Relevance

The use of self-etching systems revealed similar bond strengths to both permanent and deciduous dentin and established equivalent bond to the use of the total-etching, self-priming system on the same substrate.

SUMMARY

***Purpose*: To evaluate the bond strength of a total-etching, self-priming system (Single Bond) and a self-etching system (Clearfil SE Bond) to deciduous and permanent human dentin. *Methods and Materials*: Buccal dentin discs were prepared with a diamond disc from permanent first premolars, permanent third molars and deciduous second molars. The flat dentin surfaces were obtained by polishing with wet 600 grit silicon carbide papers. The specimens of each group were further divided into two groups for bonding to either Single Bond or Clearfil SE Bond. After 24 hours, the micro-shear bond strength testing was executed on a universal testing machine. Statistical analysis was performed at α=0.05. *Results*: No significant differences in bond strength were found between materials. However, deciduous dentin demonstrated significantly lower bond strengths than permanent premolar dentin when Single Bond was applied (p<0.05). *Conclusion*: The difference in bonding substrate (permanent or deciduous dentin) had a significant effect on bond strength when the total-etching, self-priming system was applied.**

*Pisol Senawongse, DDS, MSc, PhD, Cariology and Operative Dentistry, Department of Restorative Science, Tokyo Medical and Dental University, Japan, and Lecturer, Department of Operative Dentistry, Faculty of Dentistry, Mahidol University, Thailand

Choltacha Harnirattisai, DDS, PhD, associate professor, Department of Operative Dentistry, Faculty of Dentistry, Mahidol University, Thailand

Yasushi Shimada, DDS, PhD, lecturer, Cariology and Operative Dentistry, Department of Restorative Science, Tokyo Medical and Dental University, Japan

Junji Tagami, DDS, PhD, professor and head, Cariology and Operative Dentistry, Department of Restorative Science, Tokyo Medical and Dental University, Japan

*Reprint request: 6 Yothi Street, Phayathai, Bangkok 10400, Thailand; e-mail: dtpse@mahidol.ac.th

INTRODUCTION

Currently, two kinds of two-step adhesive resins have been developed. They are a total-etching, self-priming adhesive system and a self-etching system. The total-etching, self-priming adhesive system is composed of a self-priming adhesive resin and a separate etching agent. The wet bonding technique, bonded to wet dentin surfaces after phosphoric acid etching, is the essential requirement of this system. The self-etching system is another two-step adhesive system that is applied directly to the cavity wall without the need for a separate acid-etching step, thus liminating water rinsing and drying steps (Watanabe, Nakabayashi & Pashley, 1994; Van Meerbeek & others, 1998).

Many studies have reported differences in morphological and chemical dentin substrates between deciduous

dentin and permanent dentin (Hirayama, Yamada & Miake, 1986; Avery, 1987; Hirayama, 1990; Koutsi & others, 1994) which might effect the bond strength of adhesive systems (Mitchem & Gronas, 1986; Salama & Tao, 1991; Bordin-Aykroyd, Sefton & Davies, 1992). A number of recent comparative studies of two-step adhesive systems have investigated the application on permanent dentin (Swift & Bayne, 1997; Lucena-Martin & others, 1999; Al-Ehaideb & Mohammed, 2000), whereas, few studies with deciduous dentin existed (Bordin-Aykroyd & others, 1992; El-Kalla & García-Godoy, 1998; Burrow, Nopnakeepong & Phrukkanon, 2002). Limitation in the size of deciduous teeth seemed to be the main problem to performing these investigations.

Since the micro-shear test was developed by Shimada (Shimada & others, 2002), the evaluation of bond strength, especially on small, flat surfaces such as deciduous dentin surfaces, became possible.

This study evaluated the micro-shear bond strength of current two-step adhesive systems on deciduous dentin compared with permanent dentin.

METHODS AND MATERIALS

Two commercially available adhesive materials were used in this study. One is a total-etching, self-priming system (Single Bond, 3M, St Paul, MN, USA); the other is a self-etching system (Clearfil SE Bond, Kuraray Co, Osaka, Japan). Table 1 features details of the composition of the materials and information on the manufacturers.

Micro-shear Bond Strength Test

Labial dentin discs approximately 1-mm thick were prepared with a diamond disc (Isomet, Buehler, IL, USA) from 24 extracted permanent human first premolars, 24 extracted permanent human third molars and 24 extracted deciduous human second molars. The flat buccal dentin surfaces were obtained from the mid-coronal region by polishing with #600 grit wet silicon carbide papers. Depth of the dentin surfaces for bond testing was also controlled using the outer third of the dentin. The specimens of each substrate were further divided into two subgroups and bonded to Single Bond or Clearfil SE Bond following the manufacturers' instruction. The following steps were employed for Single Bond: etching with Scotchbond Etchant for 15 seconds, rinsing for 15 seconds, blot-drying with absorbent paper for excessive water removal leaving the surfaces moist, applying two coats of adhesive resin and light curing for 10 seconds. The steps for the Clearfil SE Bond were applying and leaving Clearfil SE Bond Primer on the surfaces for 20 seconds, drying the primed surfaces, applying Clearfil SE Bond adhesive resin and light curing for 10 seconds. Prior to the light-curing step, a plastic tube (Tygon, Norton Performance Plastic Co, Cleveland, OH, USA) 0.8-mm in diameter and 1.0-mm thick was placed on the uncured adhesive surfaces. These surfaces were then polymerized to stabilize the plastic tube with a halogen lamp curing unit (Tokuso Power Lite, Tokuyama, Tokyo, Japan). After curing, a resin composite (Clearfil AP-X, Kuraray Co, Osaka, Japan) was then placed into the tube and cured for 40 seconds. In this manner, a small cylinder of resin composite 0.8-mm in diameter and 1-mm in height was bonded to the dentin surfaces. The bonded specimens were kept in saline at 37°C for 24 hours and the plastic tubes were removed before testing. Figure 1 shows the diagram of specimen preparation.

Before testing, the specimens were checked under a stereomicroscope. Specimens that presented a defect at

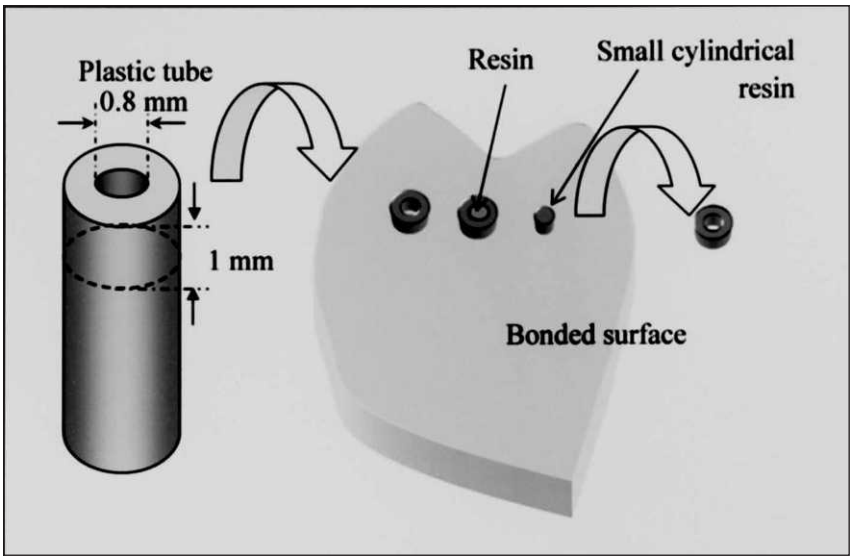


Figure 1. *Diagram of specimen preparation for micro-shear bond strength test.*

Table 1: *Details of Materials in This Study*

Products	Manufacturer	Batch #	Composition
Single Bond	3M-ESPE St Paul, MN, USA	Etch: 7432 Bonding: 3411	35% phosphoric acid HEMA, polyalkenoic acid Copolymer, Bis-GMA, photoinitiator
Clearfil SE Bond	Kuraray Osaka, Japan	Primer: 00146A Bonding: 00114A	HEMA, MDP HEMA, MDP, microfiller, photoinitiator

HEMA : 2-hydroxyethyl methacrylate
Bis-GMA : bis-phenol A diglycidylmethacrylate
MDP : 10-methacryloyloxydecyl dihydrogen phosphate

the bonding interfaces, such as interfacial gap formation or bubble inclusion, were excluded from this study. The tooth slice with a proper resin cylinder was bonded to the testing device (Bencor-Multi-T, Danville Engineering Co, San Ramon, CA, USA) which was placed in a universal testing machine (EZ test, Shimazu Ltd, Kyoto, Japan) with cyanomethacrylate glue (Zapit, Dental Ventures of America, Corona, CA, USA). A thin wire was looped around the resin cylinder and gently held flush against the resin/dentin interface. A shear force was applied at a crosshead speed of 1.0 mm/minute to failure. There were 20 specimens in each group. The data was converted to MPa unit and analyzed with two-way ANOVA and LSD multiple comparisons at 95% confidence interval.

After testing, the debonded specimens were observed under a confocal laser scanning microscope (1LM21 series, Lasertec Corporation, Tokyo, Japan). The percentages of failure were noted for the following criteria: cohesive failure in dentin, cohesive failure in resin and adhesive failure. The differences in failure modes among the groups was analyzed using the non-parametric statistical analysis and the Kruskal-Wallis one-way analysis of variance by ranks at a 0.05 level of significance.

Scanning Electron Microscopic Study of the Resin/Dentin Interface

Ten flat buccal dentin discs of extracted permanent premolars, permanent molars and deciduous second molars were utilized with the same methods as previously mentioned. The specimens for each substrate were divided into two groups and bonded with either Single Bond or Clearfil SE Bond according to instructions. The resin composites were applied to the cured bonded surfaces in a small layer about 1-2-mm thick and cured for 40 seconds. The specimens were kept in a saline solution for 24 hours after bonding.

The specimens were sectioned perpendicular to the bonded interface into approximately two equal halves and embedded in epoxy resins. The embedded specimens were polished with wet silicon carbide papers (600, 800, 1000, 1200 and 1500 grit) and diamond pastes (6, 3, 1 and 0.25 µm) sequentially. The specimens were then observed under a scanning electron microscope (JSM-5310V, JEOL Ltd, Tokyo, Japan) after argon ion beam etching (EIS-1E, Elionix Ltd, Tokyo, Japan) (Inokoshi & others, 1993) and gold sputter coating (SC-701AT Quick Coater, Elionix Ltd, Tokyo, Japan).

RESULTS

Table 2 presents the means and standard deviation in MPa of micro-shear bond strength. No significant differences were found between the bond strength of Single Bond and Clearfil SE Bond on each substrate (p=0.656). Deciduous dentin demonstrated significantly lower bond strength than permanent premolar dentin when Single Bond was applied (p=0.015). However, no significant difference in shear bond strength was demonstrated between permanent molars and deciduous dentin.

The percentages of failure are presented in Table 3. No statistically significant differences in failures were found

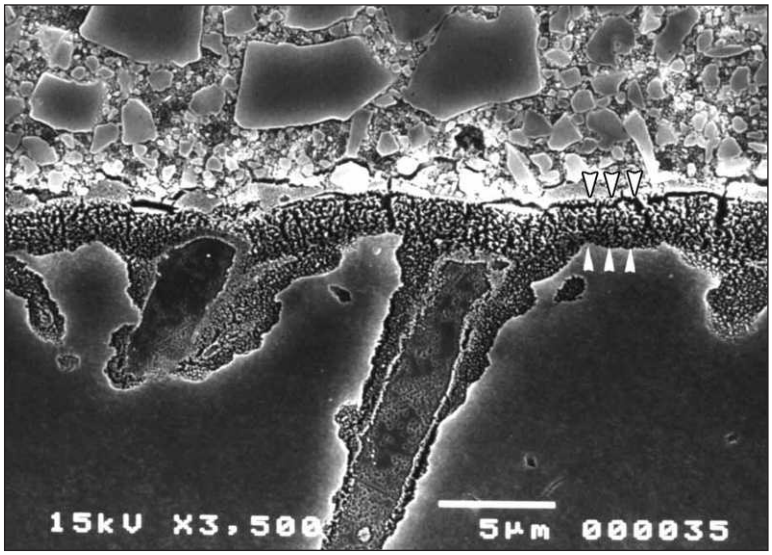


Figure 2. *SEM photograph of hybrid layer on permanent premolar dentin using Single Bond. The areas between the arrows represent the hybrid layer.*

Table 2: *Means and SD of Microshear Bond Strength on Deciduous and Permanent Dentin*

Deciduous	Permanent Teeth		Deciduous Molars
	Premolars	Molars	
Clearfil SE Bond	41.28±7.23[a,d]	39.81±6.80[a,e]	37.38±6.28[a,f]
Single Bond	41.95±9.99[b,d]	39.10±7.24[b,c,e]	35.61±6.13[c,f]

Data with the same letter present no statistically significant difference.

Table 3: *Percentages of Failure on Decidous and Permanent Dentin*

Deciduous	Permanent Teeth				Deciduous Molars	
	Premolars		Molars			
	SE	SB	SE	SB	SE	SB
Cohesive in dentin	26.75	33.50	12.00	33.50	19.00	24.25
Adhesive	73.25	66.50	88.00	66.50	81.00	75.75
Cohesive in resin	0	0	0	0	0	0

SE : Clearfil SE Bond
SB : Single Bond

between materials and among dentin substrates ($p>0.05$).

SEM observation revealed that the use of Single Bond resulted in the formation of 2-3 µm thick hybrid layers with permanent dentin (Figures 2 and 3) and deciduous dentin (Figure 4). On the other hand, about 0.5-1 µm-thick hybrid layers were found with both permanent dentin (Figures 5 and 6) and deciduous dentin (Figure 7) in the case of Clearfil SE Bond.

DISCUSSION

When the total-etching, self-priming system was applied, bond strengths to deciduous dentin were significantly lower than to permanent premolar dentin, which was in line with other investigations (Salama & Tao, 1991; Bordin-Aykroyd & others, 1992). It has been reported that the bond strengths of adhesive systems depend on the amount of solid dentin—dentin with more intertubular area (Mitchem & Gronas, 1986; Suzuki & Finger, 1988). Bond strength decreases as the pulp is approached. To gain an adequate flat surface for micro-shear testing, the deciduous dentin discs were prepared at a depth slightly closer to the pulp compared with the permanent dentin discs. This might have an effect on reducing the bond strength to deciduous dentin. In addition, the use of phosphoric acid is included in the total-etching, self-priming system. The reason for using phosphoric acid is to remove the smear layer and smear plug, as well as partial demineralization of intertubular dentin and peritubular dentin to expose the collagen mesh. This is a necessary step for the bonding mechanism of this system (Van Meerbeek & others, 1998; 2001). The acid tends to remove the peritubular dentin that was observed to be about two to five times thicker in deciduous dentin compared to permanent dentin (Hirayama & others, 1986; Avery, 1987; Hirayama, 1990; Koutsi & others, 1994). This intertubular dentin is more mineralized but less crystalline than intertubular dentin (Shellis, 1981), which would result in an increase in the width of tubules and a decrease in micro-mechanical retention.

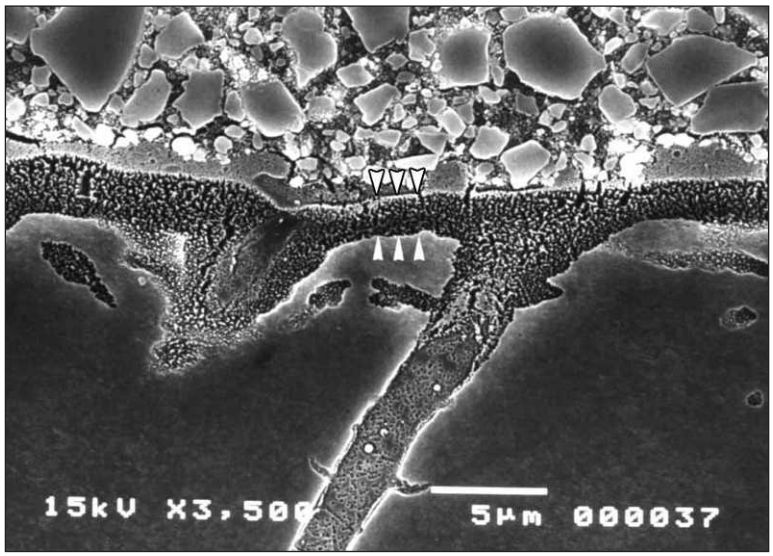


Figure 3. *SEM photograph of hybrid layer on permanent molar dentin using Single Bond. The areas between the arrows represent the hybrid layer.*

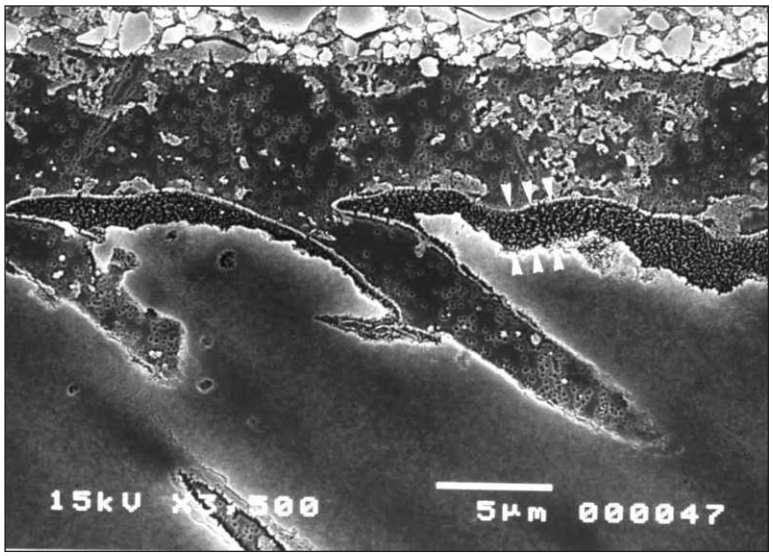


Figure 4. *SEM photograph of hybrid layer on deciduous molar dentin using Single Bond. The areas between the arrows represent the hybrid layer.*

When the self-etching system was applied, no significant difference in bond strength between permanent and deciduous dentin was observed. This was in line with a previous study (Burrow & others, 2002). The different bonding mechanisms of this system when compared with a total-etching, self-priming system (Van Meerbeek & others, 1998; 2001) might effect bond strength differently. The ability of acidic primer in a self-etching system has been reported to remove the smear layer, and partially demineralized dentin was found to be less than phosphoric acid in total-etching, self-priming systems (Van Meerbeek & others, 2001). The smear plug and undemineralized, peritubular dentin might still be present after applying acidic monomer (Van Meerbeek & others, 2001). The penetration of resin monomer into the collagen mesh of intertubular dentin might be an important factor for bonding to dentin with this system. The different amounts of peritubular dentin (Hirayama & others, 1986) might have no effect on reducing the bond strength to deciduous dentin in this situation. The bond strength of Clearfil SE Bond to deciduous dentin in this study corresponds to a previous study (Agostini, Kaaden & Powers, 2001) which demonstrated the significantly higher bond strength of Clearfil SE Bond compared to other self-etching systems on deciduous dentin.

There were no significant differences in bond strengths of both adhesive systems to permanent premolar and permanent molar dentin. Since the permanent teeth, premolars and molars were extracted for orthodontic reasons in patients 18-25 years of age, the various forms of dentin associated with physiological aging (Mjör, 1972) might be excluded. The effect of tooth position of posterior permanent teeth to bond strength could not be detected in these study conditions.

In general, the bond strength values to deciduous dentin were lower than to permanent dentin; however, only the difference between deciduous dentin and permanent premolar dentin was significant. This was the same trend as noted in previous studies (Salama & Tao, 1991; Bordin-Aykroyd & others, 1992; El-Kalla & García-Godoy, 1998; Burrow & others, 2002). The bond strengths that were obtained in this study were higher than that of other studies. This might be a consequence of using the micro-shear testing method. When conducting the micro-shear test, the trimming step that might cause reduction of bond strength is not necessary (Shimada & others, 2002).

Since the percentage of failures revealed that most failures occurred at the resin/dentin interfaces without any significant difference in materials and among teeth substrates, the prospect of failure at the resin/dentin interfaces might depend on adhesive strength.

Because of the difference in size between deciduous teeth and permanent teeth (Avery, 1987), the cavity size of deciduous teeth should be smaller than permanent teeth. Even though the results demonstrated that bonding to deciduous dentin was lower than bonding to permanent dentin, the lower bond strength with deciduous dentin might be considered clinically acceptable. The small cavities might create lower polymerization shrinkage of resin composite (Suliman, Boyer & Lakes, 1993); however, it is not clear how much bond strength should be enough to ensure a durable bond between the cavity walls and composite resin restorations, especially in the case of deciduous dentition (Davidson, de Gee & Feilzer, 1984). Further study, especially for clinical research in deciduous dentition, may be necessary to confirm the longevity of current dentin bonding systems.

For adequate bonding, current total-etching, self-priming systems require moistened dentin substrates to maintain a proper collagen mesh on etched dentin surfaces to ensure good penetration of resin monomers into this mesh. Under these circumstances, the moisture may be incidental moisture contamination from leakage of air/water syringe or saliva. This contamination is not an acceptable method of creating moist dentin, because it has been shown to discourage proper bonding (Plasmans & others, 1993; Burrow & others, 1995; Powell, Johnson & Gordon, 1995). Because of the difficulty in keeping adequate moisture and avoiding contamination in younger patients, self-etching systems may provide better results than those obtained with self-priming systems.

According to SEM observation, the action of acid conditioner of a total-etching, self-priming system to deciduous dentin seemed to be the same intensity as to permanent dentin. They demonstrated the same thickness of hybrid layer, both on deciduous dentin and on per-

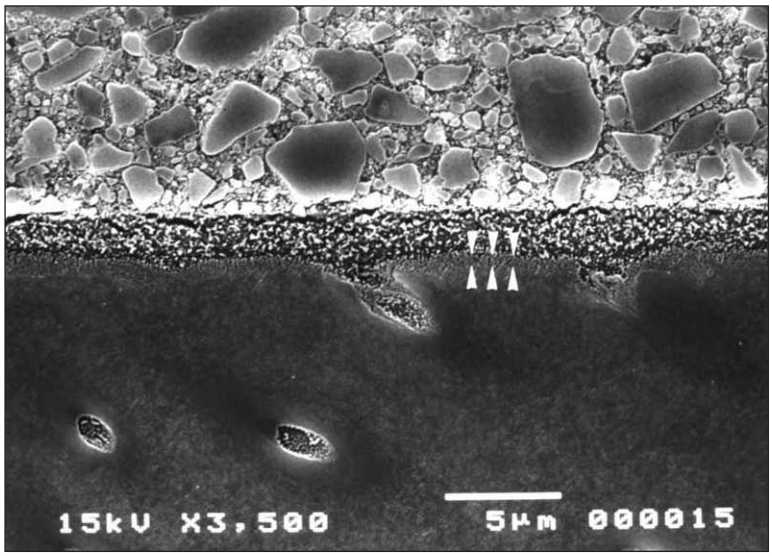


Figure 5. *SEM photograph of hybrid layer on permanent premolar dentin using Clearfil SE Bond. The areas between the arrows represent the hybrid layer.*

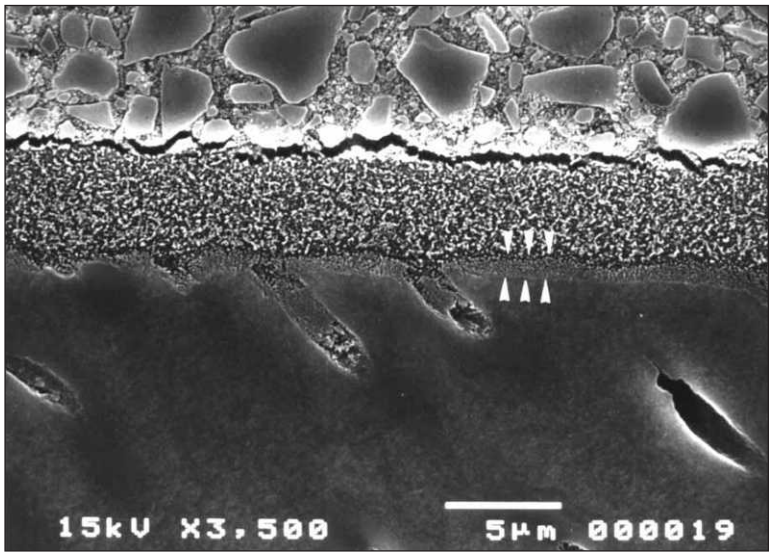


Figure 6. *SEM photograph of hybrid layer on permanent molar dentin using Clearfil SE Bond. The areas between the arrows represent the hybrid layer.*

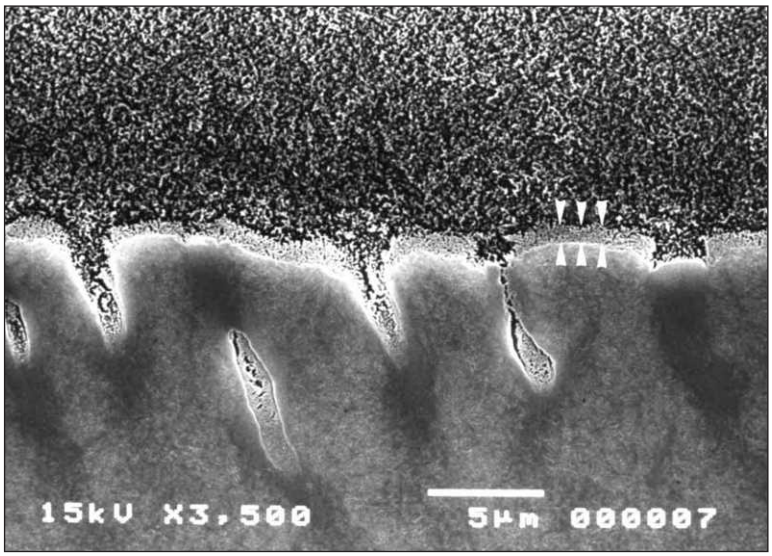


Figure 7. *SEM photograph of hybrid layer on deciduous molar dentin using Clearfil SE Bond. The areas between the arrows represent the hybrid layer.*

manent dentin, which agreed with a previous study (Burrow & others, 2002). However, this result disagreed with previous studies (Nör & others, 1996; Olmez & others, 1998). Even though the difference in mineral concentration between deciduous dentin and permanent dentin was reported (Hirayama, 1990), this might have no effect on the thickness of the hybrid layer on both deciduous and permanent dentin.

The difference in hybrid layer thickness on dentin between the application of a total-etching, self-priming system and a self-etching system might have some clinical relevance. It has been reported that the hybrid layer is an important structure for absorbing the stress that occurs from resin composite polymerization shrinkage (Van Meerbeek & others, 1993; Perdigão & others, 1996). The thicker hybrid layer might involve demineralized dentin without the penetration of adhesive resin at the bottom of this layer, which has a risk of leakage (Tam & Pilliar, 1994; Sano & others, 1994). It was reported that the difference in hybrid layer thickness might have no effect on bond strength in accordance with previous studies (Yoshiyama & others, 1996; Prati & others, 1998; Perdigão & others, 2000). The effect of a thicker hybrid layer when the total-etching self-priming system is applied and a thinner hybrid layer when the self-etching system is applied requires further study to confirm the durability of bond, especially in the clinical situation.

CONCLUSIONS

No significant differences in micro-shear bond strength between the self-etching system and the total-etching self-priming system were found. However, when the total-etching, self-priming system was applied, micro-shear bond strength was significantly lower on deciduous dentin than permanent dentin.

(Received 5 March 2003)

References

Agostini FG, Kaaden C & Powers JM (2001) Bond strength of self-etching primers to enamel and dentin of primary teeth *Pediatric Dentistry* **23(6)** 481-486.

Al-Ehaideb A & Mohammed H (2000) Shear bond strength of "one bottle" dentin adhesives *Journal of Prosthetic Dentistry* **84(4)** 408-412.

Avery JK (1987) *Oral Development and Histology* 5th ed Baltimore, Williams and Walkins Baltimore 180-190.

Bordin-Aykroyd S, Sefton J & Davies EH (1992) *In vitro* bond strengths of three current dentin adhesives to primary and permanent teeth *Dental Materials* **8(2)** 74-78.

Burrow MF, Taniguchi Y, Nikaido T, Satoh M, Inai N, Tagami J & Takatsu T (1995) Influence of temperature and relative humidity on early bond strengths to dentin *Journal of Dentistry* **23(1)** 41-45.

Burrow MF, Nopnakeepong U & Phrukkanon S (2002) A comparison of microtensile bond strengths of several dentin bonding systems to primary and permanent dentine *Dental Materials* **18(3)** 239-245.

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.

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

Hirayama A, Yamada M & Miake K (1986) An electron microscopic study of dentinal tubules of human deciduous teeth *Shika-Gakuho* **86(6)** 1021-1031.

Hirayama A (1990) Experimental analytical microscopic studies on the quantitative analysis of elemental concentrations in biological thin specimens and its application to dental science *Shika-Gakuho* **90(8)** 1019-1036.

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.

Koutsi V, Noonan RG, Horner JA, Simpson MD, Matthews WG & Pashley DH (1994) The effect of dentin depth on the permeability and ultrastructure of primary molars *Journal of Pediatric Dentistry* **16(1)** 29-35.

Lucena-Martin C, González-Rodriguez MP, Ferrer-Luque CM, Robles-Gijón V & Navajas JM (1999) Study of the shear bond strength of five one-component adhesives under stimulated pulpal pressure *Operative Dentistry* **24(2)** 73-80.

Mitchem JC & Gronas DG (1986) Effects of time after extraction and depth of dentin on resin dentin adhesives *Journal of the American Dental Association* **113(2)** 285-287.

Mjör IA (1972) Human coronal dentine: Structure and reactions *Oral Surgery, Oral Medicine, and Oral Pathology* **33**(5) 810-823.

Nör JE, Feigal RJ, Dennison JB & Edwards CA (1996) Dentin bonding: SEM comparison of the resin-dentin interface in primary and permanent teeth *Journal of Dental Research* **75(6)** 1396-1403.

Olmez A, Oztas N, Basak F & Erdal S (1998) Comparison of the resin-dentin interface in primary and permanent teeth *Journal of Clinical Pediatric Dentistry* **22(4)** 293-298.

Perdigão J, Lambrechts P, Van Meerbeek B, Tome AR, Vanherle G & Lopes AB (1996) 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, May KN Jr, Wilder AD Jr & Lopes M (2000) The effect of depth of dentin demineralization on bond strengths and morphology of the hybrid layer *Operative Dentistry* **25(3)** 186-194.

Plasmans PJ, Reukers EA, Vollenbrock-Kuipers L & Vollenbrock HR (1993) Air humidity: A detrimental factor in dentine adhesion *Journal of Dentistry* **21(4)** 228-233.

Powell LV, Johnson GH & Gordon GE (1995) Factors associated with clinical success of cervical abrasion/erosion restorations *Operative Dentistry* **20(1)** 7-13.

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

Salama FS & Tao L (1991) Comparison of gluma bond strength to primary versus permanent teeth *Journal of Pediatric Dentistry* **13(3)** 163-166.

Sano H, Shono T, Takatsu T & Hosoda H (1994) Microporous dentin zone beneath resin-impregnated layer *Operative Dentistry* **19(2)** 59-64.

Shellis RP (1981) Dental tissues in Osborn JW editor *Dental Anatomy and Embryology* Oxford Blackwell Scientific Publications 155-210.

Shimada Y, Senawongse P, Harnirattisai C, Burrow MF, Nakaoki Y & Tagami J (2002) Bond strength of two adhesive systems to primary and permanent enamel *Operative Dentistry* **27(4)** 403-409.

Suliman AA, Boyer DB & Lakes RS (1993) Cusp movement in premolars resulting from composite polymerization shrinkage *Dental Materials* **9(1)** 6-10.

Suzuki T & Finger WJ (1988) Dentin adhesives: Site of dentin versus bonding of composite resins *Dental Materials* **4(6)** 379-383.

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

Tam LE & Pilliar RM (1994) Effects of dentin surface treatments on the fracture toughness and tensile bond strength of a dentin-composite adhesive interface *Journal of Dental Research* **73(9)** 1530-1538.

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

Van Meerbeek B, Perdigão J, Lambrechts P & Vanherle G (1998) The clinical performance of adhesives *Journal of Dentistry* **26(1)** 1-20.

Van Meerbeek B, Vargas M, Inoue S, Yoshida Y, Peumans M, Lambrechts P & Vanherle G (2001) Adhesive and cements to promote preservation dentistry *Operative Dentistry* **(Supplement 6)** 119-144.

Watanabe I, Nakabayashi N & Pashley DH (1994) Bonding to ground dentin by a phenyl-P self-etching primer *Journal of Dental Research* **73(6)** 1212-1220.

Yoshiyama M, Carvalho RM, 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, 2004, **29-2**, 203-211

The Effect of One-Step Polishing System on the Surface Roughness of Three Esthetic Resin Composite Materials

LS Türkün • M Türkün

Clinical Relevance

The PoGo one-step micro-polisher can be used safely to polish anterior resin composite restorations with a reduced time application.

SUMMARY

Proper finishing of restorations is desirable not only for aesthetic considerations but also for oral health. The primary goal of finishing is to obtain a restoration that has good contour, occlusion, healthy embrasure forms and a smooth surface. This study investigated: 1) analyzing the surface roughness of three resin composites finished and polished with a new one-step and two conventional multi-step polishing systems and 2) evaluating the effectiveness of one-step polishing system and surface morphology using scanning electron microscope analysis (SEM).

Specimens (n=72) measuring 8-mm in diameter x 2-mm in thickness were fabricated in a plexiglass mold covered with a Mylar strip using three esthetic resin composites. After polymerization, six specimens per resin composite received no finishing treatment and served as a control. Fifty-four specimens were randomly polished with Sof-Lex discs, Enhance disc with polishing paste or PoGo for 30 seconds after being ground wet with a 1200 grit silicon carbide paper. The average surface roughness of each polished specimen was determined with a profilometer (Surtronic 4). The data were analyzed using repeated measures ANOVA and Scheffe's post-hoc test of multiple comparisons ($p \leq 0.01$). Representative samples of the mentioned finishing procedures were selected and examined using a scanning electron microscope (SEM).

There was no surface roughness in all resin composites tested against Mylar strip. The results showed no difference between the surfaces of Clearfil ST and Esthet-X polished with PoGo and the Mylar group ($p \geq 0.01$). Among all the polishing systems tested, PoGo exhibited the smoothest finish for all resin composites. The combination of Enhance and Prisma Gloss polishing paste exhibited the highest roughness values for Filtek A110 and Clearfil ST; however, it gave the same Ra values as PoGo for Esthet-X ($p \leq 0.01$).

*L Sebnem Türkün, DDS, PhD, assistant professor, Ege University School of Dentistry, Department of Restorative Dentistry and Endodontics, Izmir- Turkey

Murat Türkün, DDS, PhD, associate professor, Ege University School of Dentistry, Department of Restorative Dentistry and Endodontics, Izmir- Turkey

*Reprint request: 35100 Izmir/Turkey; e-mail: sebnemturkun@hotmail.com

SEM analysis of Esthet-X samples confirmed the profilometer's results. The surfaces of the Clearfil ST discs polished with PoGo resemble that of Mylar, while Enhance and Sof-Lex exposed and dislodged the filler particles. PoGo scratched in some places Filtek A110's surface, while Enhance produced mostly a Mylar-like surface with dislodged fillers in some places.

INTRODUCTION

The continuous development of aesthetically acceptable adhesive restorative materials has made a variety of tooth-colored materials available for clinical use. Currently, the clinician has resin composite, polyacid-modified composite, resin-modified glass ionomer and traditional glass ionomer restoratives as options for direct restorations. In addition, resin composite materials are available with a variety of filler types that affect both their handling characteristics and physical properties. The ultimate aesthetics of these tooth-colored restoratives is strongly influenced by the final surface polish (Yap, Lye & Sau, 1997; Pratten & Johnson, 1988; Bouvier, Duprez & Lissac, 1997; Jung 2002), and smooth, highly polished restorations have been shown to be more aesthetic and more easily maintained than restorations with rougher surfaces (Strassler & Bauman, 1993; Hachiya & others, 1984; Stanfod & others, 1985).

Early studies have shown that the smoothest surface of a resin restoration is attained when the resin is polymerized against an appropriate matrix strip. When a matrix is not used, polymerization of the outer layer is inhibited, resulting in a surface layer rich in organic binder with stick and soft consistency. In either case, removal of that outermost resin by trimming and finishing procedures would lead to producing a harder, more wear resistant, and, hence, a more aesthetically stable surface.

Proper finishing of restorations is desirable not only for aesthetic considerations but also for oral health. The primary goal of finishing is to obtain a restoration with good contour, occlusion, healthy embrasure forms and a smooth surface. Tight margins should blend aesthetically into the tooth's natural contours, and resin composite restorations should be smooth so as to reduce plaque retention and minimize possible gingival irritation, surface staining, patient discomfort and recurrent decay (Weitman & Eames, 1975; Chan, Fuller & Hormati, 1980; Fruits, Miranda & Coury, 1996; Bollen, Lambrechts & Quirynen, 1997; Neme & others, 2002; MacCabe & Caddick, 1978; Brunsuold & Lane, 1990). The trimming procedure for resin-based restorations comprises four steps:

1. **Coarse Finishing or Reduction of Excess:** Instruments with high grinding effectiveness are preferred but, due to the coarse abrasiveness, they should only be used on restorative material.

2. **Contouring:** The aim is to achieve final form of the restoration as prescribed by functional and aesthetic criteria.

3. **Fine Finishing:** This comprises the final, precise adjustment of restoration margins and improvement in surface smoothness.

4. **Polishing:** A smooth and glossy, but nonetheless textured surface is the final objective of any polishing procedure.

Clinicians have their choice among a wide range of finishing and polishing instruments. The most popular include diamond or carbide burs, stones, rubber wheels-cups and points, discs, strips and pastes.

Previously, most of the emphasis was placed on the application of progressively finer grits of abrasives to polish resin composite restorations. Today, many attempts have been made to develop composite finishing instruments that are suitable for all four steps of the trimming procedure (Fruits & others, 1996). A set of highly flexible polyurethane-based finishing and polishing discs coated with aluminum oxide particles are widely used. More recently, a disposable diamond micro-polisher disc has been introduced with the aim of achieving all four trimming procedures using only one instrument.

This investigation 1) analyzed the surface roughness of three aesthetic resin composites finished and polished with a new one-step and two conventional multi-step polishing systems and 2) evaluated the effectiveness of the one-step polishing system and its possible surface damage by scanning electron microscope (SEM) analysis.

METHODS AND MATERIALS

Three light-cured resin composites intended for anterior aesthetic restorations were used in this study. The resin composites evaluated were Clearfil ST (Kuraray Europe GmbH, Düsseldorf, D-40549, Germany), Filtek A110 (3M Dental Product, St Paul, MN, USA) and Esthet-X (Dentsply/Caulk, Milford, DE, USA). Table 1 shows the properties of the materials.

The finishing and polishing systems tested were Sof-Lex discs (3M Dental Products), Enhance Polishing system with polishing pastes (Dentsply/Caulk) and PoGo one-step diamond coated micro polisher (Dentsply/Caulk). Table 2 shows the components of the polishing systems tested.

Using a plexiglass mold (Plexiglass MC, Rohm ve Haas, Philadelphia, PA, USA), 8x2 mm disc specimens were prepared. For each resin composite, 24 discs were fabricated and a total of 72 discs was obtained. Six specimens per resin composite received no finishing treatment after

being cured under Mylar strips (SS White Co, Philadelphia, PA, USA) and served as a control.

The resin composites were placed using a plastic instrument and covered with a Mylar strip. A glass slide 1-2 mm thick was placed over the strip before curing with the light-activating source (Degulux/Degussa, Frankfurt/Main 11, Germany) to flatten the surfaces. The samples were then cured for 60 seconds through the Mylar strip and the glass slide. Output of the light was checked using a photometric tester (Dentek, Inc, Buffalo, NY, USA) so as to be in excess of 400mW/cm^2. The curing light guide of the light-curing unit was moved on both sides of the specimen for an additional 20 seconds after removing the strips and glasses. The cured samples were then stored in 100% humidity at 37°C for 24 hours prior to finishing procedures.

After storage, the Mylar-created surfaces were evaluated with a profilometer (Surtronic 4, Taylor Hobson Ltd, Denmark) on a flat plane to obtain average surface roughness values and surface profile tracings that would serve as a baseline for the polishing systems. The remaining 54 samples were ground wet with a 1200 grit silicon carbide paper on a metallurgical finishing wheel to provide a baseline, then polished with the previously mentioned systems. To reduce variability, the same investigator polished the surfaces using the same slow-speed handpiece (approximately 20,000 rpm) with a constantly moving repetitive stroking action to prevent heat build up and the formation of grooves. The Mylar strips, foam instruments and abrasive discs were discarded after each use.

For the first group, Sof-Lex Pop-On discs at medium, fine and super-fine grits were used for 30 seconds each on the composite samples. After each step of polishing, all specimens were thoroughly rinsed with water and air dried before the next step until final polishing. The Enhance finishing discs were applied 30 seconds for the intermediate finishing and a foam-polishing cup with Prisma Gloss fine and super fine polishing paste were applied 30 seconds for the second group. The last group was polished with the flat, broad surface of the PoGo diamond micro-polisher disc and first applied with light intermittent pressure, then with decreased pressure to increase the surface luster using a light buffing motion for 30 seconds. The Mylar strip surfaces of each resin composite served as a control.

The polished resin composite discs were washed, allowed to dry and kept in 100% humidity for 24 hours before measuring the average surface roughness (Ra). Five successive measurements in different directions were recorded for the six specimens in each group.

The average surface roughness (Ra) was the major parameter reported, representing the arithmetic mean of the height of all surface irregularities over a predetermined linear segment (0.40 mm) of each specimen. This feature is commonly referred to as surface finish. All readings were made with the instrument range selection set at 0.00 to 2.00 µm and the cut-off selector set at 0.8 mm. The sensitive diamond-tipped stylus arm, which has a preset stylus force of 2mN, was used and the mean values for Ra were determined for each group.

Statistical differences were checked by ANOVA. When differences were found between groups, Scheffe's post-hoc test of multiple comparisons ($p \leq 0.01$) was used.

One representative specimen of each group was prepared for the scanning electron microscope (JEOL JSM

Table 1: *The Composition, Manufacturers and Batch Numbers of the Resin Composites Tested*

Resin Composites	Composition	Manufacturers	Batch #s
Clearfil ST (microfilled)	Colloidal silica, borosilicate glass (0.04 µ, 81.6%) BisGMA, TEGDMA	Kuraray Europe GmbH, Düsseldorf, Germany	00019A
Filtek A110 (microfilled)	Colloidal silica (0.04 µ, 56%) BisGMA, TEGDMA	3M Dental Products St Paul, MN, USA	2BP
Esthet-X (micro-hybrid)	Bariumalumino fluoroborosilicate glass (BAFG) and nano-sized silicon dioxide particles (0.7µ, 77%) Modified BisGMA, TEGDMA, UDMA	Dentsply/Caulk, Milford, DE, USA	001206

Table 2: *The Composition and Batch Numbers of the Polishing Systems Investigated*

Polishing Systems	Composition	Manufacturers	Batch #s
Sof-Lex Pop-On Discs	Medium aluminum oxide disc (40 µm) Fine aluminum oxide disc (24 µm) Ultra-fine aluminum oxide disc (8 µm)	3M Dental Products, St Paul, MN, USA	P020403
Enhance disc with polishing paste	Aluminum oxide disc (40 µm) Fine aluminum oxide paste (1 µm) Extra-fine aluminum oxide paste (0.3 µm)	Dentsply/Caulk, Milford, DE, USA	001228 001031
PoGo	Diamond coated micro-polisher	Dentsply/Caulk, Milford, DE, USA	020409

5200, Tokyo, Japan). Specimens were sputter coated with gold to a thickness of approximately 200Å in a vacuum evaporator. Photographs of representative areas of the polished surfaces were taken at 500x magnifications.

RESULTS

The average surface roughness and standard deviation values produced by the Mylar strips, Sof-Lex discs, Enhance/Paste systems and PoGo on the three resin composite materials are listed in Table 3 and Figure 1.

The smoothest surfaces for all the resin composites tested were obtained against the Mylar strip. No statistically significant differences were observed among them ($p \geq 0.01$). The results showed no statistically significant difference between the surfaces of the Clearfil ST and Esthet-X samples polished with the PoGo system and the control Mylar group ($p \geq 0.01$). Among all the polishing systems tested, PoGo exhibited the smoothest finish for all resin composites. The combination of the Enhance system and Prisma Gloss polishing paste exhibited the highest roughness values for Filtek A110 and Clearfil ST; however, it gives the same Ra values as PoGo for Esthet-X ($p \leq 0.01$).

SEM analysis of the Esthet-X samples polished with PoGo revealed the same surface appearance as the Mylar strip, while the surfaces polished with Enhance had some scratches. The Sof-Lex discs wore the resin matrix, exposing the filler particles (Figure 2 a-d). The surfaces of the Clearfil ST discs polished with PoGo resemble that of Mylar, while Enhance and Sof-Lex exposed and dislodged the filler particles (Figure 3 a-d). PoGo scratched in some places Filtek A110's surface, while Enhance produced mostly a Mylar-like surface with dislodged fillers in some places. The Sof-Lex system scratched and exposed fillers of the Filtek A110 resin composite (Figure 4 a-d).

DISCUSSION

A resin composite restoration can be imperceptible to the naked eye when its surface closely resembles the surrounding enamel surface. Thus, polished restorations should demonstrate an enamel-like surface texture and gloss. Previous studies (Dennison, Fan & Powers, 1981; Wilson, Heath & Watts, 1990) have shown that the smoothest obtainable surface on resin composite restorations is achieved by curing the material in direct contact with a smooth matrix surface (Mylar strip). Since such a finish cannot be maintained,

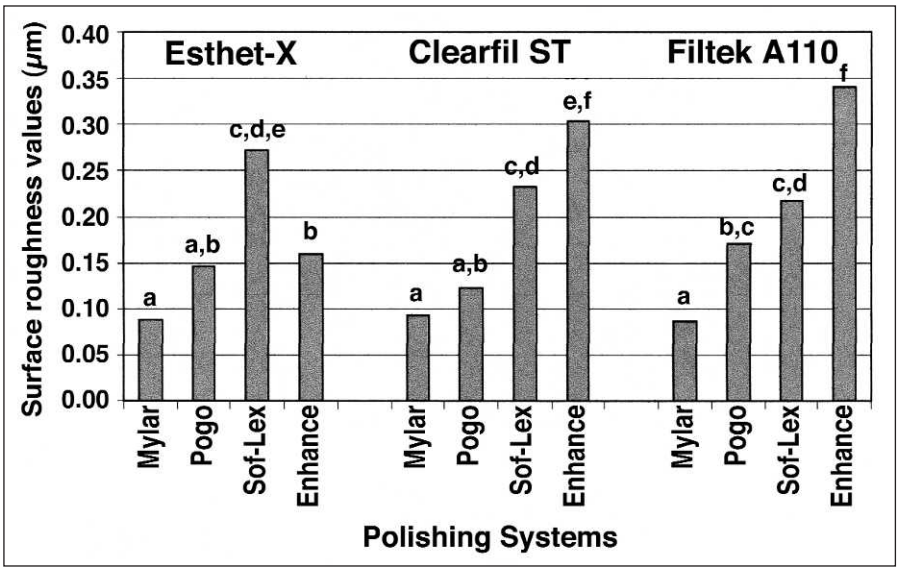


Figure 1. *Mean surface roughness values for the resin composites tested. There was no significant difference with the bars labeled with the same letters (p<0.01).*

Table 3: *Mean Ra Values (μm), Standard Deviations and Standard Errors for the Various Materials and Polishing Systems Evaluated*

Restorative Materials	Polishing Systems	n	Mean Ra Values	Standard Deviation	Standard Error
Esthet-X	*Mylar*	6	0.088 μm	0.140	0.004
	PoGo	6	0.146 μm	0.039	0.011
	Sof-Lex	6	0.273 μm	0.041	0.012
	Enhance	6	0.159 μm	0.022	0.006
Clearfil ST	*Mylar*	6	0.093 μm	0.012	0.004
	PoGo	6	0.122 μm	0.039	0.011
	Sof-Lex	6	0.232 μm	0.044	0.013
	Enhance	6	0.303 μm	0.075	0.022
Filtek A110	*Mylar*	6	0.087 μm	0.021	0.006
	PoGo	6	0.171 μm	0.030	0.009
	Sof-Lex	6	0.217 μm	0.028	0.008
	Enhance	6	0.346 μm	0.101	0.029

further contouring and finishing are required. It is clinically important to determine the finishing technique that results in the smoothest surface with minimum time and instruments.

For years, specially designed diamonds with very fine abrasive particle size and white Arkansas stones have been used to polish resin composite restorations (Roulet, Hirt & Lutz, 1984). However, the use of diamond burs are limited to initial contouring because of their ability to remove equal amounts of adjacent enamel (Quiroz & Lentz, 1986). Later, most of the emphasis was placed on the application of progressively finer grits of abrasives to polish resin composites with little concern for the types of motion employed during their use. Various motions may be equally critical to the development of optimal surface smoothness. A rotary motion (circular), a planar motion and a reciprocating motion can be employed to polish the surface of resin composites. In rotary motion (diamonds and cylindrical stones), the axis of rotation of the abrasive device is parallel to the surface being smoothed. The planar motion is a rotational movement with the axis of rotation of the abrasive device perpendicular to the surface being smoothed (abrasive discs). The reciprocating motion is employed when a finishing strip is pulled back and forth over a surface (Fruits & others, 1996).

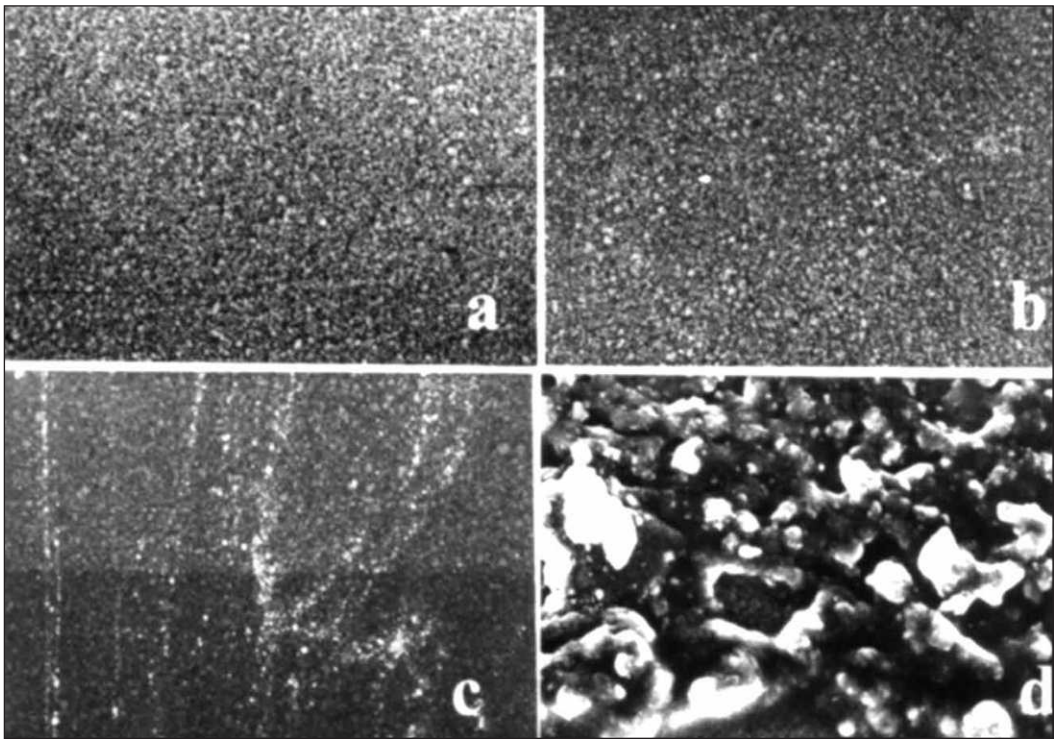


Figure 2: *Esthet-X surfaces polished with different systems (500x magnification).* Figure 2*a. Control surface (Mylar); b. Polished with PoGo; c. Polished with Enhance; d. Polished with Sof-Lex.*

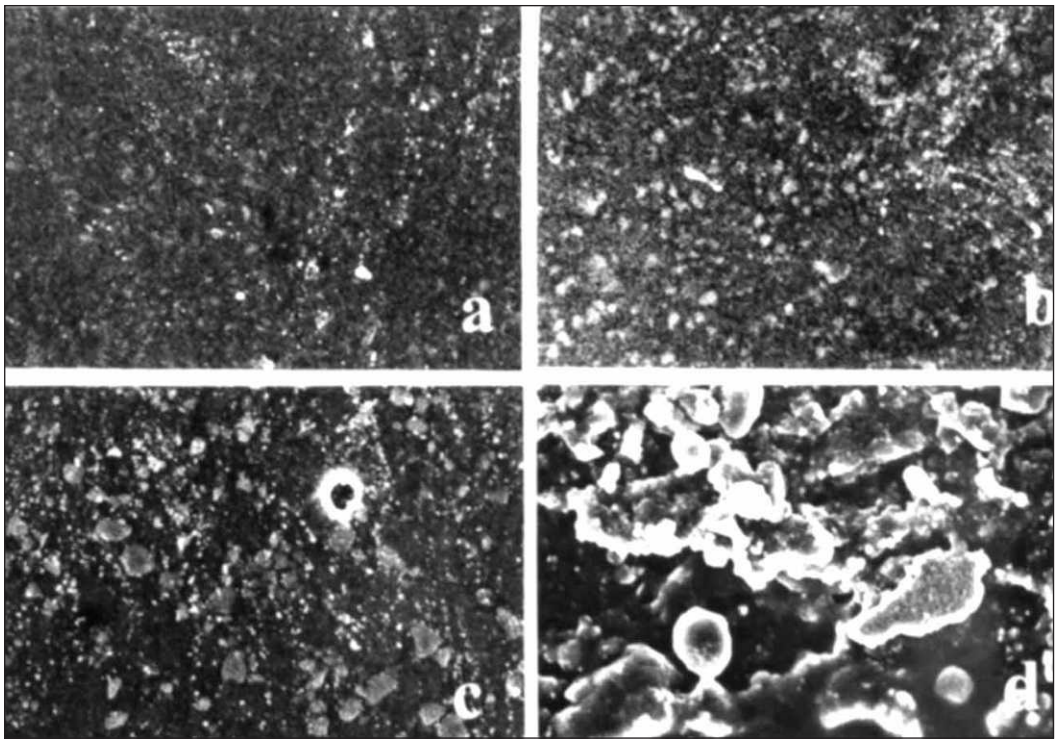


Figure 3: *Clearfil ST surfaces polished with different systems (500x magnification).*Figure 3*a. Control surface (Mylar); b. Polished with PoGo; c. Polished with Enhance; d. Polished with Sof-Lex.*

The results obtained by Fruits and others (1996) comparing different polishing motions on restorative materials showed that for all possible combinations of materials and abrasive grits, the planar motion achieved the lowest average roughness values. In this study, all the systems were tested using a planar motion.

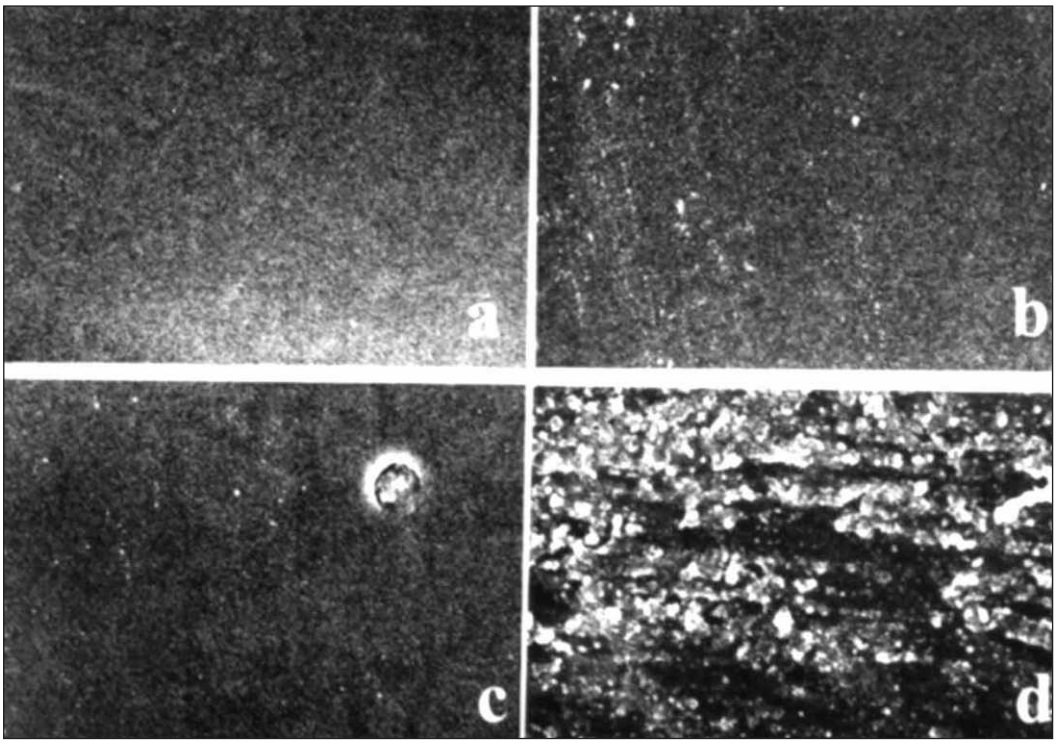


Figure 4: *Filtek A110 surfaces polished with different systems (500x magnifications).* Figure 4*a. Control surface (Mylar); b. Polished with PoGo; c. Polished with Enhance; d. Polished with Sof-Lex.*

For years, flexible discs with progressively finer grits of abrasives have been efficient for finishing and polishing resin composites (Hoelscher & others, 1998; Tate, De Schepper & Cody, 1992; Setcos, Tarim & Suzuki, 1999). Several studies stated that the large particles embedded in Sof-Lex discs tend to rip though the surface of the resin composites and, when used with certain hybrid composites, tend to cut and abrade filler particles and resin matrix equally, resulting in a smooth surface (VanNoort & Davis, 1984; Chen, Chan & Chan, 1988; Van Dijken & Ruyter, 1987). Unfortunately, their use is confined to convex surfaces, thus, necessitating the use of alternative techniques for anatomically structured surfaces such as the occlusal surface of posterior teeth or the lingual aspect of anterior teeth. Rigid rotary instruments such as diamonds and tungsten carbide burs have been recommended for finishing, using either a single rotary instrument or a sequence of burs. Rubber polishers, felt wheels and different pastes are used to polish structured surfaces (Northeast & VanNoort, 1988). More recently, PoGo, a new diamond micro-polisher was introduced to reduce the steps and time necessary to polish resin composites.

Even though the effects of previous finishing instruments on the surface roughness of resin composites have been well studied, the results are controversial (Dennison & others, 1981; Lutz, Setcos & Phillips, 1983). This difference is partly attributed to the size, hardness and amount of filler of the resins used to restore the teeth. The smoothest surfaces can be obtained with anterior resin materials having a higher percentage of resin and submicron particles. When surface roughness is evaluated, another contributing variable is the resin composite system used. When conventional and hybrid composites are finished with stones or tungsten carbide burs, the macrofillers are more likely to be dislodged than ground down, causing detectable surface irregularities. It has been generally accepted that conventional resin composites produce rougher surfaces than those that are microfilled (Craig & others, 2002).

The inherent surface roughness of a restoration must be equal to or lower than the surface roughness of enamel on enamel-to-enamel occlusal contact areas (Ra=0.64 µm) (Willems & others, 1991). Although the Mylar strip control group provided the lowest values, the surfaces produced were not without surface imperfections (Ra=0 µm). This may be explained by the fact that any surface imperfections in the Mylar strip will be reproduced in the surface of restoratives (VanNoort, 1983). Although this type of finish may be the surface treatment of choice, clinical situations may require bulk removal of excess composite and gross trimming followed by a polishing system. In this study, Mylar strips, as expected, produced the smoothest surface with a roughness average of 0.09 µm. However, the new diamond micro-polisher PoGo produced comparable surface roughness (0.17 to 0.12 µm) for all resin composites tested with only one disc and a short application time of 30 seconds. Sof-Lex discs produced the second smoothest surfaces for Clearfil ST and Filtek A110. The use of polishing paste does not appear to be necessary to improve the finish obtained using abrasive impregnated discs if clinical significances are considered.

However, Enhance discs and polishing pastes seem to produce better surface smoothness for Esthet-X compared to Sof-Lex. According to the current study, surface profile analysis showed that smoothness was not dependent on the use of a systematic series of instruments and polishing materials characterized by decreasing abrasive particle size.

Stoddard and Johnson (1991) suggested that, because of the variations in filler particles and types of resin, it is important to pair a resin composite with a matching polishing system. Additional factors affecting polishing results may include the amount of pressure utilized, the orientation of the abrading surface and the amount of time spent with each abrasive material. In this study, Enhance discs with polishing pastes and PoGo seem to produce better surface smoothness for Esthet-X. This can be attributed to the fact that all products are from the same company and may be more compatible with each other.

In their study, Hergott, Ziemiecki and Dennison (1989) found that the surface roughness values produced by the Sof-Lex disc system were statistically similar to those produced against the Mylar strips. In this study, PoGo exhibited comparable surface smoothness to the Mylar strips for Esthet-X and Clearfil ST.

According to Marigo and others (2001), the final glossy surface obtained by polishing depended on the flexibility of the backing materials in which the abrasive is embedded, the hardness of the particles, the instruments and their geometry. In this study, the diamond coated flexible micro-polisher disc produced the glossiest surface among all the polishing systems tested. Furthermore, it demonstrated that using polishing paste following abrasive impregnated finishing discs did not greatly improve the surface smoothness of the composite materials. This study agrees with the results by Marigo and others (2001) and Chen and others (1988).

Tate and Powers (1996) evaluated the surface roughness of composites and hybrid ionomers. According to their findings, both the Enhance and Sof-Lex finishing and polishing systems produced smoother surfaces than the 12-fluted finishing bur applied to the surface of specimens for the resin composites tested. Furthermore, they found that aluminum oxide discs appeared to finish the materials without dislodging the glass particles. However, according to the results of this study, the Enhance system produced rougher surfaces than Sof-Lex for Clearfil ST and Filtek A110.

Profilometers have been used for years to measure surface roughness for *in vitro* investigations. They provide limited two-dimensional information, but an arithmetic average roughness can be calculated and used to represent various material/polishing surface combinations that assist clinicians in their treatment decisions (McLundie & Murray, 1974; MacCabe & Caddick, 1978). However, the complex structure of a surface cannot be fully characterized by the use of only surface roughness measurements. Therefore, it is not appropriate to draw conclusions on the clinical suitability of a finishing instrument exclusively based on roughness average results. However, in combination with a SEM analysis that permits an evaluation on the destructive potential of a finishing tool, more valid predictions of clinical performance can be made (Lambrechts & Vanherle, 1982; McLundie & Murray, 1974). In this study, surface roughness measurements were used only for relative comparisons. In addition, the respective injury that can be produced by using the new finishing instrument PoGo was analyzed with the SEM (Marigo & others, 2001; Krejci, Lutz & Boretti, 1999). As observed, the results of the profilometric measurements were largely confirmed by SEM analysis.

The question as to which degree of a surface must be finished cannot be answered sufficiently. Achieving the roughness of occluding enamel surfaces (Willems & others, 1991) or achieving profile irregularities smaller than the average size of bacteria (Shintani & others, 1985) is discussed as possible thresholds. According to Chung (1994), restorations appear optically smooth when their surface roughness is smaller than 1 µm. In this study, all tested systems were found to be effective in polishing the resin composites tested.

CONCLUSIONS

Considering the reduced steps, application time, elimination of cross-infection risks and achievement of Mylar strip-like surfaces, PoGo diamond micro-polisher can be the polishing system to be used in anterior resin composites. According to the good SEM and profilometer results obtained, the authors can anticipate that PoGo will be an innovation in the field of polishing systems.

However, caution must be exercised when interpreting the results of *in vitro* studies. Correlation to clinical practice may be limited to situations where accessible, relatively flat surfaces are finished because most of the newest polishing systems are in a disc shape. Further studies are needed to determine which finishing technique is best suited to clinical situations, where access is limited and restoration surfaces are not flat.

Acknowledgements

The manufacturers generously provided the resin composites and polishing systems tested in this study. The authors express their sincere appreciation to Dr BH Sen for his assistance with the Scanning Electron Microscope analysis of this investigation.

Note

This study was presented as an oral presentation in the 81st IADR meeting in Göteborg/Sweden (24-28 June 2003).

(Received 6 March 2003)

References

Bollen CML, Lambrechts P & Quirynen M (1997) Comparison of surface roughness of oral hard materials to the threshold surface roughness for bacterial plaque retention: A review of the literature *Dental Materials* **13(4)** 258-269.

Bouvier D, Duprez JP & Lissac M (1997) Comparative evaluation of polishing systems on the surface of three aesthetic materials *Journal of Oral Rehabilitation* **24(12)** 888-894.

Chan KC, Fuller JL & Hormati AA (1980) The ability of foods to stain two composite resins *Journal of Prosthetic Dentistry* **43(5)** 542-545.

Chen RC, Chan DC & Chan KC (1988) A quantitative study of finishing and polishing techniques for a composite *Journal of Prosthetic Dentistry* **59(3)** 292-297.

Chung KH (1994) Effects of finishing and polishing procedures on the surface texture of resin composites *Dental Materials* **10(5)** 325-330.

Craig RG, Hanks CT, Kohn DH, Koran III A, O'Brien WJ, Powers JM, Wapner WC & Wataha JC (2002) *Restorative Dental Materials* 11th ed St Louis CV Mosby p 226, 238.

Dennison JB, Fan PL & Powers JM (1981) Surface roughness of microfilled composites *Journal of the American Dental Association* **102(6)** 859-862.

Fruits TJ, Miranda FJ & Coury TL (1996) Effects of equivalent abrasive grit sizes utilizing differing polishing motions on selected restorative materials *Quintessence International* **27(4)** 279-285.

Hachiya Y, Iwaku M, Hosoda H & Fusayama T (1984) Relation of finish to discoloration of composite resins *Journal of Prosthetic Dentistry* **52(6)** 811-814.

Hergott AM, Ziemiecki TL & Dennison JB (1989) An evaluation of different composite resin systems finished with various abrasives *Journal of the American Dental Association* **119(6)** 729-732.

Hoelscher DC, Neme AM, Pink FE & Hughes PJ (1998) The effect of three finishing systems on four esthetic restorative materials *Operative Dentistry* **23(1)** 36-42.

Jung M (2002) Finishing and polishing of a hybrid composite and a heat-pressed glass ceramic *Operative Dentistry* **27(2)** 175-183.

Krejci I, Lutz F & Boretti R (1999) Resin composite polishing—Filling the gaps *Quintessence International* **30(7)** 490-495.

Lambrechts P & Vanherle G (1982) Observation and comparison of polished composite surfaces with the aid of SEM and profilometer *Journal of Oral Rehabilitation* **9(2)** 169-182.

Lutz F, Setcos JC & Phillips RW (1983) New finishing instruments for composite resins *Journal of the American Dental Association* **107(4)** 575-580.

MacCabe JF & Caddick RJ (1978) The finishing of composite restorations *British Dental Journal* **145(4)** 101-104.

Marigo L, Rizzi M, LaTorre G & Rumi G (2001) 3-D surface profile analysis: Different finishing methods for resin composites *Operative Dentistry* **26(6)** 562-568.

McLundie AC & Murray FD (1974) Comparison of methods used in finishing composite resin—a scanning electron microscope study *Journal of Prosthetic Dentistry* **31(2)** 163-171.

Neme AL, Frazier KB, Roeder LB & Debner TL (2002) Effect of prophylactic polishing protocols on the surface roughness of esthetic restorative materials *Operative Dentistry* **27(1)** 50-58.

Northeast SE & VanNoort R (1988) Surface characteristics of finished posterior composite resins *Dental Materials* **4(5)** 278-288.

Pratten DH & Johnson GH (1988) An evaluation of finishing instruments for an anterior and posterior composite *Journal of Prosthetic Dentistry* **60(2)** 154-158.

Quiroz L & Lentz DL (1986) The effect of polishing procedures on the surface smoothness of several light-cured posterior composites *Compendium* **7(9)** 676-678.

Roulet JF, Hirt T & Lutz F (1984) Surface roughness and marginal behavior of experimental and commercial composites an *in vitro* study *Journal of Oral Rehabilitation* **11(5)** 499-509.

Setcos JC, Tarim B & Suzuki S (1999) Surface finish produced on resin composites by new finishing systems *Quintessence International* **30(3)** 169-173.

Shintani H, Satou J, Satou N, Hayashihara H & Inoue T (1985) s finishing methods on staining and accumulation of *Streptococcus mutans* HS-6 on composite resins *Dental Materials* **1(6)** 225-227.

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

Strassler HE & Bauman G (1993) Current concepts in polishing composite resins *Practical Periodontics Aesthetic Dentistry* **5(3 Supplement 1)** 12-17.

Tate WH, DeSchepper EJ & Cody T (1992) Quantitative analysis of six composite polishing techniques on a hybrid composite material *Journal of Esthetic Dentistry* **4 Supplement** 30-32.

Tate WH & Powers JM (1996) Surface roughness of composites and hybrid ionomers *Operative Dentistry* **21(2)** 53-58.

Van Dijken JW & Ruyter IE (1987) Surface characteristics of posterior composites after polishing and toothbrushing *Acta Odontologica Scandinavia* **45(5)** 337-346.

VanNoort R (1983) Controversial aspects of composite resin restorative materials *British Dental Journal* **155(11)** 380-385.

VanNoort R & Davis LG (1984) The surface finish of composite resin restorative materials *British Dental Journal* **157(10)** 360-364.

Weitman RT & Eames WB (1975) Plaque accumulation on composite surfaces after various finishing procedures *Journal of the American Dental Association* **91(1)** 101-106.

Willems G, Lambrechts P, Braem M, Vuylsteke-Wauters M & Vanherle G (1991) The surface roughness of enamel-to-enamel contact areas compared with the intrinsic roughness of dental resin composites *Journal of Dental Research* **70(9)** 1299-1305.

Wilson F, Heath JR & Watts DC (1990) Finishing composite restorative materials *Journal of Oral Rehabilitation* **17(1)** 79-87.

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

©Operative Dentistry, 2004, **29-2,** 212-218

Correlation Between Microleakage and Cement Thickness in Three Class II Inlay Ceramic Systems

W Romão, Jr • WG Miranda, Jr
PF Cesar • RR Braga

Clinical Relevance

Inlays built with heat-pressed and CAD-CAM ceramic systems presented lower cement thickness and less microleakage in dentin compared to sintered inlays.

SUMMARY

The objectives of this study include comparing the cement thickness and microleakage of Class II ceramic inlays built with three ceramic systems and verifying whether there was a correlation between those two variables. The ceramic systems used include: 1) Heat-pressed (IPS-Empress); 2) CAD-CAM (CEREC 2) and 3) Sintered (Colorlogic). Standardized MOD Class II inlay cavities with one proximal box extending below and the other extending above the cement-enamel junction (CEJ) were prepared in 30 extracted human molars and randomly assigned to three groups. The ceramic inlays were constructed according to manufacturer's instructions and cemented using a dual-cure resin cement (Variolink II). All teeth were mechanically cycled (100,000 cycles, 78N) and thermocycled (700 cycles, 5°C-55°C). After immersion in silver nitrate, the inlays were sectioned mesial-distally and evaluated with an optical microscope (40x). The cement thickness obtained by the Colorlogic system (enamel: 113 ± 25 µm; dentin: 118 ± 23 µm) was significantly higher than that obtained by CEREC (enamel: 78 ± 14 µm; dentin: 87 ± 13 µm) and Empress (enamel: 65 ± 15 µm; dentin: 89 ± 14 µm). Regarding dye penetration, there was no statistical difference among the three ceramic systems in enamel. At the dentin margins, the Colorlogic system resulted in a significantly higher penetration depth compared to CEREC and Empress, which had similar average values. No correlation was found between cement thickness and microleakage either in enamel or dentin for any of the ceramic systems.

Waldyr Romão, Júnior, DDS, MS, graduate student, Department of Dental Materials, University of São Paulo, Brazil

Walter Gomes Miranda, Júnior, DDS, MS, PhD, assistant professor, Department of Dental Materials, University of São Paulo, Brazil

*Paulo Francisco Cesar, DDS, MS, PhD, assistant professor, Department of Dental Materials, University of São Paulo, Brazil

Roberto Ruggiero Braga, DDS, MS, PhD, assistant professor, Department of Dental Materials, University of São Paulo, Brazil

*Reprint request: Av Prof Lineu Prestes, 2227 – Cidade Universitária "Armando Salles de Oliveira," São Paulo – SP – Brasil, CEP: 05508-900; e-mail: paulofc@usp.br

INTRODUCTION

Problems associated with microleakage in ceramic inlays such as marginal discoloration, post-operative

sensitivity and recurrent caries are often reported in clinical trials. The overall poor fit of the restorations is claimed to be the main cause related to those occurrences (Fuzzi & others, 1991; Aberg, van Dijken & Olofsson, 1994; Gemalmaz & others, 1997; Lim & Ironside, 1997; Audenino & others, 1999). Marginal discoloration over time is one of the most common findings and has been reported in up to 55% of the restorations after two years (Molin & Karlsson, 2000). Post-operative sensitivity has also been a matter of concern and varies from 0% to 13% (Molin & Karlsson, 2000; Pallesen & van Dijken, 2000; Frankenberger, Petschelt & Krämer, 2000). With respect to the occurrence of secondary caries, some clinical studies show no incidence (Thordrup, Isidor & Hörsted-Bindslev, 1999; Pallesen & van Dijken, 2000; Molin & Karlsson, 2000), but other researchers have reported an incidence from 2% to 8% between four and eight years (Isidor & Brondum, 1995; Fradeani, Aquilano & Bassein, 1997; Hayashi & others, 2000a).

In order to achieve restorations with an improved marginal adaptation, a variety of ceramic systems have been developed as an alternative to the powder-liquid systems. These new technologies include the computer-designed restorations (CAD-CAM) and heat-pressed ceramics. The CEREC system (Sirona Dental Systems, Bensheim, Germany, D 64625) was introduced into dentistry in the early 1980s and three versions have already been developed (Sturdevant, Bayne & Heymann, 1999). While in the original system (CEREC 1), the restoration was milled with only one tool; in the second version (CEREC 2), the milling chamber with a disk and a diamond-coated cylinder allows for a more accurate restoration (Mörmann & Schug, 1997; Parsell & others, 2000). Recently, a third version of the system (CEREC 3) was released. The main improvement in this new version is the presence of two diamond-coated cylinders as cutting tools in the milling chamber (Mörmann & Bindl, 2000).

Heat-pressed ceramic systems use a leucite-based ceramic ingot pressed at 1150°C under 0.3 to 0.4 MPa into a refractory mold formed by the lost wax technique (Denry, 1996). Adaptation of the inlays obtained by this system have proven to be better than what was obtained with fired inlays (Sulaiman & others, 1997; Audenino & others, 1999; Beschnidt & Strub, 1999; Ferrari & others, 1999; Hahn & others, 2000; Hahn & others, 2001) mainly because few dimensional changes occur during the pressing of the ceramic into the investment mold. Similar to metal casting techniques, the contraction that occurs during cooling is compensated for by expansion of the investment material (Kelly, Nishimura & Campbell, 1996).

In the literature, there are studies that compare the microleakage and/or precision of fit of heat-pressed, CAD-CAM and sintered porcelains systems (Dietschi, Maeder & Holz, 1992; Krejci, Lutz & Reimer, 1993; Eskander & Shehab, 1994; Sjögren, 1995; Sulaiman & others, 1997; Audenino & others, 1999; Beschnidt & Strub, 1999). With respect to the precision of fit, Audenino and others (1999) found that a heat-pressed system (Empress) showed mean gap values of 53 µm, which was significantly less than the mean value of 85 µm found with a fired inlay system (Colorlogic). Sjögren (1995) compared the marginal and internal fit of a CAD-CAM system (CEREC 1) and a heat-pressed system (Empress) and found that the computer-machined system had the poorest adaptation. Regarding microleakage, Eskander and Shehab (1994) found no differences in the degree of dye penetration between a CAD-CAM (CEREC 1) and a fired system (Vitadur N). Hahn and others (2001) studied the influence of precision fit on the microleakage of ceramic inlays and found that the variation of gap values from 27 µm to 406 µm had no effect on the degree of microleakage when a highly viscous resin cement was used, but when a low viscosity cement was used, the microleakage was higher in thicker luting spaces.

Considering that there is little information on the relationship between microleakage and adaptation of ceramic inlays, this study compared the cement line thickness and microleakage of Class II inlays built with three ceramic systems and verified whether there was a correlation between the two variables. The null hypothesis was that there is a correlation between microleakage and cement thickness in bonded ceramic inlays.

METHODS AND MATERIALS

Thirty caries-free, recently extracted human third molars were selected for this study. After extraction, the teeth were cleaned using scalers to remove soft tissue remnants and stored in 0.9% saline solution at room temperature for up to four weeks prior to preparation. Standardized mesio-occlusodistal Class II preparations were performed using a six-degree conical high-speed diamond bur (N 2131, KG Sorensen, Barueri, Brazil) with air-water spray. Preparations were made as follows: the buccolingual width of the proximal and occlusal boxes was 2 mm, and the depth of the pulpal and axial walls was 2 mm. A butt-joint margin preparation was made at the cervical margins, with no bevels. All internal angles were rounded. One of the proximal boxes had the cervical margin placed in enamel 2 mm above the cementum-enamel junction (CEJ). The opposite proximal box was extended 1 mm below the CEJ to place the cervical margin in dentin. The preparation was finished with a fine-grained bur (N 1090, KG Sorensen). The prepared teeth were randomly assigned to three experimental groups corresponding to the three ceramic systems tested. Colorlogic (Dentsply Ind e Com, Rio de Janeiro, Brazil)

is a powder-liquid feldspathic porcelain system used with a refractory die technique. IPS-Empress (Ivoclar, Schaan, Liechenstein) uses high-leucite ceramic ingots that are heat-pressed into an investment mold obtained by a lost-wax casting technique. CEREC 2 (Sirona, Bensheim, Germany) is a chairside CAD-CAM system, where the inlay is machined onto fine-grained feldspathic porcelains blocks (Vitablocks Mark II, Vita, Germany). All the systems tested used shade A3 ceramic.

Impressions of all teeth were taken using a polyvinylsiloxane-based material (Aquasil, Dentsply), except for those restored with the CEREC 2 system. A double-mixing technique (low and high consistency in separate steps) was performed using a 20-mm diameter, 15-mm high perforated PVC tube as tray. The impressions were poured with extra hard type IV die stone (Durone, Dentsply).

In the first group (Colorlogic), the stone dies were duplicated with refractory die material (Dentsply) using the same impression material described above. The refractory dies underwent firing at 920°C for 10 minutes in a ceramic oven (Phoenix, Dentsply) to avoid water absorption from the porcelain during the inlay build up. A die spacer (Yeti Dental, Germany) was then applied on the cavity walls of the refractory die without reaching the margins. Porcelain slurry was applied to the refractory die using the brush technique. The porcelain was submitted to two firing cycles and one autoglazing cycle following the manufacturer's recommendations. In group 2 (IPS-Empress), an inlay pattern was waxed up directly onto the stone die, invested (Empress Refractory Investment, Ivoclar) and pressure cast at 1150°C according to the manufacturer's protocol. The inlay was then finished with fine-grained diamond burs and glazed at a maximum temperature of 920°C, heating rate of 45°C/minute and no vacuum. In the third group (CEREC 2), the prepared teeth were coated with a very thin reflective film and scanned with the CEREC 2 system intraoral camera. Design of the external surfaces of the inlay was determined by CEREC 2 software.

All inlays were bonded to the prepared teeth using the same protocol. The inner surface of the restoration was etched for four minutes with 10% hydrofluoric acid (Dentsply), rinsed for one minute and dried with compressed air. Two layers of a silane solution (Monobond-S, Vivadent, Schaan, Liechenstein) were applied to the etched surface and allowed to air dry for 60 seconds. Bonding agent (Prime & Bond NT, Dentsply) was applied and light cured for 10 seconds at 600 mW/cm^2 (Optilux 400, Demetron, Danbury, CT, USA). The prepared surfaces of the teeth were cleaned with pumice in a rubber cup. Enamel margins were etched with 37% phosphoric acid for 15 seconds. Then, without rinsing the enamel, the acid was applied to the dentin surface for 10 seconds. The cavity was then thoroughly rinsed with water for 30 seconds and gently air dried to remove excess water, leaving the cavity visibly moist. Bonding agent (Prime & Bond NT, Dentsply) was applied to tooth surfaces and light cured for 10 seconds.

A dual-cure cement (Variolink II, Vivadent) was mixed according to the manufacturer's instructions (using the high viscosity catalyst) and placed on the inner surface of the inlay and cavity walls. The restoration was inserted into the cavity and kept under a constant pressure of 10 N using a surveyor. After removing the excess cement, glycerin gel (Liquid Strip, Vivadent) was applied on all margins of the restoration to avoid contact with oxygen. The composite cement was then light cured for 40 seconds from the buccal, lingual and occlusal aspects (120 seconds total). During the curing procedure, the tooth was adapted in a device that simulated the presence of the adjacent teeth.

The specimens were stored for one week in distilled water at 37°C, then subjected to 100,000 cycles of mechanical loading in water at room temperature under a maximum load of 78 N and a frequency of approximately 4 Hz. The specimens were then subjected to 700 thermal cycles in water between 5°C and 55°C, with a dwell time of 60 seconds.

The tooth surfaces were coated with two layers of nail varnish, leaving only the gingival area of both proximal boxes exposed. The specimens were then immersed in 50% silver nitrate solution for two hours in the absence of light and cleaned in tap water, dried with absorbent paper and immersed in developing solution for six hours under the light of a halogen lamp (Photoflood, GE, Stanford, CT, USA). The specimens were then sectioned through the center of the restorations in a mesial-distal direction using a low-speed diamond blade saw.

The depth of dye penetration and thickness of the cement line at the gingival margins were measured in µm using a light microscope (Zeiss, Oberkochen, Germany, 73447) under 40x magnification. The images were captured by digital camera (Sony Corporation, Tokyo, Japan) connected to a computer using image analyzer software (developed at the Department of Stomatology of the University of São Paulo). Cement thickness was measured on the sectioned halves of the cervical wall at three locations: close to the margin, in the middle of the cervical wall and close to the internal angle. Cement thickness data were analyzed by means of two-way ANOVA. Multiple comparisons were performed using Tukey's post-hoc test at a pre-set significance level of 5%. Microleakage measurements were analyzed using Kruskal-Wallis non-parametric one-way ANOVA by ranks with Bonferroni correction (significance level was defined p=0.05) due to the abnormal distribution and lack of homocedasticity (unequal vari-

ances) of the data. The correlation between cement thickness and microleakage was verified by means of Pearson's correlation test.

RESULTS

The results are shown in Table 1. The statistical analysis revealed that the cement thickness obtained by the Colorlogic system was significantly higher than that obtained by the CEREC and Empress systems, which presented statistically similar results. For the three systems, the cement thicknesses in enamel and dentin were similar.

For dye penetration data, both numerical penetration depth averages (with the respective standard deviations) and mean ranks are reported. No statistical difference was observed among the mean ranks of the ceramic systems in enamel. At the dentin margins, the Colorlogic system resulted in significantly higher mean rank than the other two systems. Regardless of the ceramic system, dentin margins always presented statistically higher dye penetration than enamel margins. Figure 1 shows the plot of microleakage versus cement thickness. No correlation was found between the two variables either in enamel or dentin. The Pearson's correlation coefficient for enamel and dentin, respectively, were -0.424 (p=0.222) and -0.005 (p=0.990) for Colorlogic, -0.100 (p=0.784) and -0.149 (p=0.681) for Cerec, and 0.017 (p=0.965) and 0.307 (p=0.388) for Empress.

DISCUSSION

In this study, the thickness of the cement line was used to estimate the fit of ceramic inlays. The fit achieved after cementation is considered one of the most relevant aspects for the long-term clinical perform-ance of indirect restorations (Alkumru & others, 1988). Even though it has been postulated that the smallest possible gap is required for indirect metallic restorations, Sjögren (1995) stated that when an adhesive inlay is considered, the ideal values of both marginal and internal adaptation have not yet been conclusively determined. One could expect that a small gap would result in a modest volume of luting composite and problems associated with the polymerization shrinkage would be reduced (Audenino & others, 1999). Also, it has been demonstrated that a small gap is necessary to reduce clinical wear of the resin cement at the margins of the restoration (Krejci & others, 1993). However, Feilzer, de Gee and Davidson (1989) showed that the stress caused by polymerization shrinkage of the luting composite in a highly confined situation (as represented by the marginal gap) is likely to surpass the bond strength between the tooth substrate and the resin cement. When a composite polymerizes in a confined setting, its shrinkage manifests as stress. It has been demonstrated that the magnitude of stress developed in bonded restorations is directly related to the ratio of the bonded-to-unbonded surface, so the called "configuration factor" (C-factor)

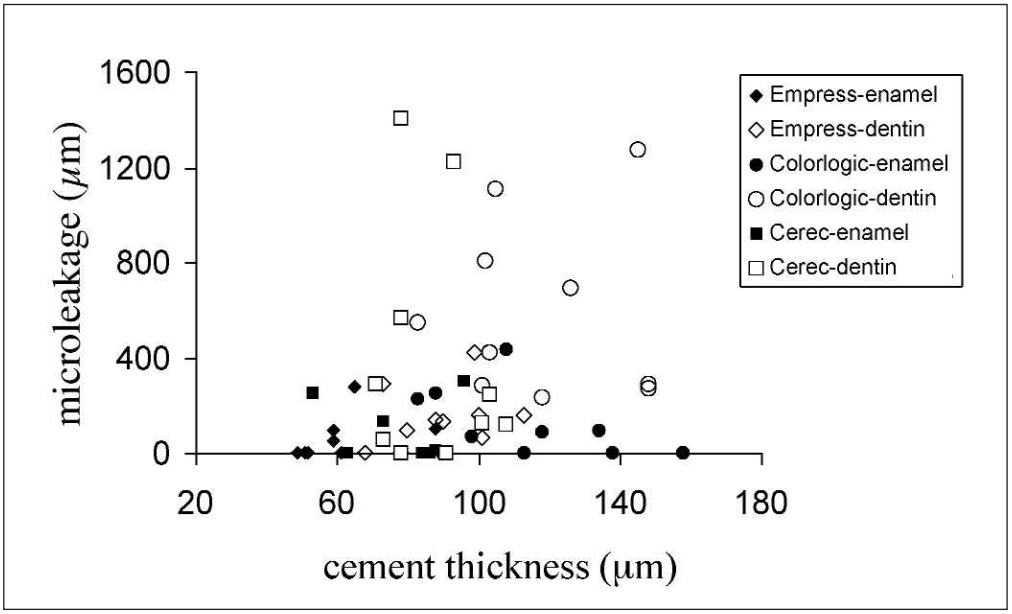


Figure 1. *Plot of microleakage versus cement thickness (both in μm).*

Table 1: *Average and Standard Deviation of Cement Thickness and Microleakage (both in μm). For Microleakage, Mean Ranks of the Kruskal-Wallis Test Are Also Provided. Values Followed by the Same Superscript Are Not Statistically Different (p>0.05)*

System	Mean Cement Thickness (μm)		Microleakage			
			Enamel		Dentin	
	Enamel	Dentin	Mean (μm)	Mean Rank Value	Mean (μm)	Mean Rank Value
Colorlogic	113 ± 25*	118 ± 23*	117 ± 145	25.3[b,c,d]	593 ± 371	50.5[a]
CEREC	78 ± 14#	87 ± 13#	70 ± 116	20.8[c,d]	404 ± 511	36.6[b]
Empress	65 ± 15#	89 ± 14#	53 ± 90	18.7[d]	146 ± 128	31.2[b,c]

(Feilzer, de Gee & Davidson, 1987). Therefore, theoretically, an inverse relationship could be expected between cement thickness and microleakage. However, within the range of cement thickness values displayed by the three ceramic systems tested, no correlation was found with microleakage. It is possible that the differences in C-factor were not great enough to influence the contraction stress significantly. In spite of the lack of statistical correlation between cement thickness and microleakage, the three ceramic systems ranked similarly on both tests. Alster and others (1997) demonstrated that in thin resin layers (varying from 50 to 2700 µm), the stress generated due to polymerization shrinkage decreases as the layer thickness increases. In the same study, however, the authors found no significant differences in contraction stress developed in layers between 50 and 200 µm. They hypothesized that polymerization contraction of a thin layer of resin cement is minimal in an absolute sense, and even a small deformation of the bonding substrate could provide significant stress relief. As the compliance provided by the tooth substrate is similar for all specimens, thinner layers of cement would have a better chance of dissipating the stress that resulted from polymerization shrinkage compared to thicker layers, which might explain the findings of the current study.

Clinically acceptable margin gap values for metallic restorations have been reported to be up to 50 to 70 µm (Löfstrom & Barakat, 1989). Ceramic restorations have shown a less precise fit compared to metallic restorations, with gap values ranging from 50 to 300 µm due to the high firing shrinkage of porcelain (Audenino & others, 1999). From the perspective of fracture mechanics, Molin, Karlsson and Kristiansen (1996) have determined that a cement thickness between 50 and 100 µm is preferable to reduce the risk of porcelain fracture. Other authors have suggested that the maximum width of exposed luting cement should not exceed 100 µm in order to reduce wear (Leinfelder, Isenberg & Essig, 1989; Van Meerbeek & others, 1992; Schmalz, Federlin & Reich, 1995).

The results of cement thickness in this study agree with previous studies that found higher marginal gap values for fired inlays compared to heat-pressed and CAD-CAM systems and a similar performance of the latter two (Sjögren, 1995; Audenino & others, 1999). The marginal gaps of CAD-CAM restorations with first-generation equipment (CEREC 1) varied from 80 to 282 µm (Sturdevant & others, 1999). The second-generation equipment (CEREC 2) was designed to minimize errors during fabrication of the restoration. In fact, the results of this study show a better adaptation for CEREC 2 (mean cement line thickness 83 ± 10 µm) when compared to the values reported for CEREC 1. Other studies that used the second-generation of the CEREC system have shown gaps varying from 56 to 97 µm (Mörmann & Schug, 1997; Sturdevant & others, 1999; Parsell & others, 2000).

The interfacial gap for the IPS Empress system has been reported to vary from 23 to 92 µm (Sulaiman & others, 1997; Audenino & others, 1999; Beschnidt & Strub, 1999; Ferrari & others, 1999; Hahn & others, 2000; Hahn & others, 2001). Sjögren (1995) also observed satisfactory marginal adaptation when comparing Empress inlays with the In-Ceram, Celay and CEREC 1 systems. The good performance of heat-pressed systems with respect to marginal adaptation might be related to the fact that most dimensional change occurs during cooling and can be controlled by the appropriate investment expansion (Kelly & others, 1996). Heat-pressed ceramics seem to be an excellent alternative as an esthetic indirect restorative material. In addition to satisfactory adaptation, this system has shown better strength and fracture toughness compared to conventional feldspatic porcelains due to the more homogeneous distribution of the crystalline leucite particles (Dong & others, 1992).

The average cement thickness obtained by Colorlogic in this study was significantly higher than the mean values of the other systems and was the only one to exceed 100 µm. The results of gap measurements usually reported for fired ceramic restorations show a wide variation. Some studies (Dietschi & others, 1992; Hayashi & others, 2000b) present gap values that vary from 28 to 57 µm, while others show values varying from 78 to 145 µm (Fuzzi & others, 1991; Aberg & others, 1994; Gemalmaz & others, 1997; Lim & Ironside, 1997; Audenino & others, 1999). A significant volumetric shrinkage occurs inherent to the firing process of conventional powder-liquid porcelains that varies from 15% to 40% depending on the porcelain used (Denry, 1996). The main reason for this volume change is the fact that sintering is done partly under vacuum, and when air at atmospheric pressure is allowed to enter the furnace (at a high temperature), it has a strong compressive effect on the semi-molten porcelain. The result is a restoration with higher density, less porosity and a smaller size (Craig, 1997). The sintering contraction of the ceramic can be compensated for by subsequent bakings along the margins of the restoration but not inside the preparation (Audenino & others, 1999). Apparently, the setting expansion of refractory die is not accurate enough to compensate for the firing shrinkage.

The results of dye penetration showed no statistical difference in microleakage at the enamel margins. Nevertheless, there was a noticeable tendency for the Colorlogic system to present higher microleakage values than the CEREC 2 and Empress systems. The reason why the statistical test found no differences between these two groups may have been the high standard deviations obtained in microleakage values.

Quantitative measurements are more accurate but result in higher dispersion of the results compared to qualitative measurement (scores from 0 to 5) (Hahn & others, 2001). Microleakage at the dentin margins was higher than the enamel margins. This is a common finding in microleakage studies (Sim & others, 1994; Christgau & others, 1999; Ferrari & others, 1999). Dentin shows a wider biologic variability than enamel, which makes it more difficult to create a strong bond that will resist the interfacial stress generated by resin cement polymerization shrinkage and also subsequent thermal and mechanical stresses (Manhart & others, 2001). At the dentin margins, the inlays made with the Colorlogic system showed significantly higher microleakage than the other two systems. It suggests that on dentin, larger cement lines may result in higher microleakage. Dye penetration at the cement-ceramic interface is not likely to occur since all the ceramic systems tested are feldspathic porcelains that provide a strong, reliable bond to composite when subjected to hydrofluoric acid etching and silanization (Della Bona & van Noort, 1998).

The viscosity of the luting cement has been proven to be another factor influencing the correlation between microleakage and precision of fit. Hahn and others (2001) found that for a highly viscous resin cement (Variolink Ultra), the variation of gap values from 27 μm to 406 μm had no effect on the degree of microleakage. However, when a low viscous cement (Variolink II) was used, the microleakage was significantly higher in larger luting spaces, which agrees with the findings of this study. The lack of statistical correlation between cement thickness and microleakage suggests that there are other factors involved in the interfacial integrity of bonded ceramic inlays. The observation of the dispersion plot (Figure 1) indicates that although the range of values for cement thickness was similar for both enamel and dentin, the range of microleakage values was higher in dentin, suggesting that bonding between dentin and the adhesive is less reproducible than in enamel and might be one of the factors involved in the interfacial integrity of the restoration.

CONCLUSIONS

In conclusion, the results of this study showed that inlays made with a heat-pressed system or a CAD-CAM system presented significantly less thickness of the cement line at the gingival wall compared to a fired porcelain system. No significant correlation was observed between cement thickness and microleakage, therefore, the null hypothesis must be rejected. However, the system with the poorest adaptation presented the highest incidence of microleakage at the dentin margin.

(Received 7 March 2003)

References

Aberg CH, van Dijken JW & Olofsson AL (1994) Three-year comparison of fired ceramic inlays cemented with composite resin or glass ionomer cement *Acta Odontologica Scandinavica* **52(3)** 140-149.

Alkumru H, Hullah WR, Marquis PM & Wilson HJ (1988) Factors affecting the fit of porcelain jacket crowns *British Dental Journal* **164(2)** 39-43.

Alster D, Feilzer AJ, de Gee AJ & Davidson CL (1997) Polymerization contraction stress in thin resin composite layers as a function of layer thickness *Dental Materials* **13(3)** 146-150.

Audenino G, Bresciano ME, Bassi F & Carossa S (1999) *In vitro* evaluation of fit of adhesively luted ceramic inlays *International Journal of Prosthodontics* **12(4)** 342-347.

Beschnidt SM & Strub JR (1999) Evaluation of the marginal accuracy of different all-ceramic crown systems after simulation in the artificial mouth *Journal of Oral Rehabilitation* **26(7)** 582-593.

Christgau M, Friedl KH, Schmalz G & Resch U (1999) Marginal adaptation of heat-pressed glass-ceramic veneers to dentin *in vitro Operative Dentistry* **24(3)** 137-146.

Craig RG (1997) *Restorative Dental Materials* (10th edition) Mosby St Louis.

Della Bona A & van Noort R (1998) Ceramic surface preparations for resin bonding *American Journal of Dentistry* **11(6)** 276-280.

Denry IL (1996) Recent advances in ceramics for dentistry *Critical Review of Oral Biology and Medicine* **7(2)** 134-143.

Dietschi D, Maeder M & Holz J (1992) *In vitro* evaluation of marginal fit and morphology of fired ceramic inlays *Quintessence International* **23(4)** 271-278.

Dong JK, Luthy H, Wohlwend A & Scharer P (1992) Heat-pressed ceramics: Technology and strength *The International Journal of Prosthodontics* **5(1)** 9-16.

Eskander ME & Shehab GI (1994) Microleakage of computer-generated Vita Cerec and Vitadur—N laminate veneers *Egyptian Dental Journal* **40(1)** 593-600.

Feilzer AJ, de Gee AJ & Davidson CL (1987) Setting stress in composite resin in relation to configuration of the restoration *Journal of Dental Research* **66(11)** 1636-1639.

Feilzer AJ, de Gee AJ & Davidson CL (1989) Increased wall-to-wall curing contraction in thin bonded resin layers *Journal of Dental Research* **68(1)** 48-50.

Ferrari M, Mason PN, Fabianelli A, Cagidiaco MC, Kugel G & Davidson CL (1999) Influence of tissue characteristics at margins on leakage of Class II indirect porcelain restorations *American Journal of Dentistry* **12(3)** 134-142.

Fradeani M, Aquilano A & Bassein L (1997) Longitudinal study of pressed glass-ceramic inlays for four and a half years *Journal of Prosthetic Dentistry* **78(4)** 346-353.

Frankenberger R, Petschelt A & Krämer N (2000) Leucite-reinforced glass ceramic inlays and onlays after six years: Clinical behavior *Operative Dentistry* **25(6)** 459-465.

Fuzzi M, Luthy H, Wohlwend A, Di Febo G, Carnevale G & Caldari R (1991) Marginal fit of three different porcelain onlays bonded to tooth: An *in vitro* study *International Journal of Periodontics and Restorative Dentistry* **11(4)** 303-316.

Gemalmaz D, Özcan M, Yoruç AB & Alkumuru HN (1997) Marginal adaptation of a sintered ceramic inlay system before and after cementation *Journal of Oral Rehabilitation* **24(9)** 646-651.

Hahn P, Attin T, Gröfke M & Hellwig E (2001) Influence of resin cement viscosity on microleakage of ceramic inlays *Dental Materials* **17(3)** 191-196.

Hahn P, Schaller H, Hafner P & Hellwig E (2000) Effect of different luting procedures on the seating of ceramic inlays *Journal of Oral Rehabilitation* **27(1)** 1-8.

Hayashi M, Miura M, Nishimura N, Takeshige F & Ebisu S (2000a) Effects of cavity form and setting expansion of refractory dies on adaptability of Class II (MO and MOD) fired ceramic inlays *Operative Dentistry* **25(6)** 549-554.

Hayashi M, Tsuchitani Y, Kawamura Y, Miura M, Takeshige F & Ebisu S (2000b) Eight-year clinical evaluation of fired ceramic inlays *Operative Dentistry* **25(6)** 473-481.

Isidor F & Brondum K (1995) A clinical evaluation of porcelain inlays *Journal of Prosthetic Dentistry* **74(2)** 140-144.

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

Krejci I, Lutz F & Reimer M (1993) Marginal adaptation and fit of adhesive ceramic inlays *Journal of Dentistry* **21(1)** 39-46.

Leinfelder KF, Isenberg BP & Essig ME (1989) A new method for generating ceramic restorations: A CAD-CAM system *Journal of the American Dental Association* **118(6)** 703-707.

Lim C & Ironside JG (1997) Grit blasting and the marginal accuracy of two ceramic veneer systems—a pilot study *Journal of Prosthetic Dentistry* **77(4)** 359-364.

Löfstrom LH & Barakat MM (1989) Scanning electron-microscopic evaluation of clinically cemented cast gold restorations *Journal of Prosthetic Dentistry* **61(6)** 664-669.

Manhart J, Schmidt M, Chen HY, Kunzelmann KH & Hickel R (2001) Marginal quality of tooth-colored restorations in Class II cavities after artificial aging *Operative Dentistry* **26(4)** 357-366.

Molin MK & Karlsson SL (2000) A randomized 5-year clinical evaluation of 3 ceramic inlay systems *International Journal of Prosthodontics* **13(3)** 194-200.

Molin MK, Karlsson SL & Kristiansen MS (1996) Influence of film thickness on joint bend strength of a ceramic/resin composite joint *Dental Materials* **12(4)** 245-249.

Mörmann WH & Bindl A (2000) The Cerec 3—a quantum leap for computer-aided restorations: Initial clinical results *Quintessence International* **31(10)** 699-712.

Mörmann WH & Schug J (1997) Grinding precision and accuracy of fit of Cerec 2 CAD-CIM inlays *Journal of the American Dental Association* **128(1)** 47-53.

Pallesen U & van Dijken JW (2000) An 8-year evaluation of sintered ceramic and glass ceramic inlays processed by the Cerec CAD/CAM system *European Journal of Oral Sciences* **108(3)** 239-246.

Parsell DE, Anderson BC, Livingston HM, Rudd JI & Tankersley JD (2000) Effect of camera angulation on adaptation of CAD/CAM restorations *Journal of Esthetic Dentistry* **12(2)** 78-84.

Schmalz G, Federlin M & Reich E (1995) Effect of dimension of luting space and luting composite on marginal adaptation of Class II ceramic inlay *Journal of Prosthetic Dentistry* **73(4)** 392-399.

Sim C, Neo J, Chua EK & Tan BY (1994) The effect of dentin bonding agents on the microleakage of porcelain veneers *Dental Materials* **10(4)** 278-281.

Sjögren G (1995) Marginal and internal fit of four different types of ceramic inlays after luting—an *in vitro* study *Acta Odontologica Scandinavica* **53(1)** 24-28.

Sturdevant JR, Bayne SC & Heymann HO (1999) Marginal gap size of ceramic inlays using second-generation CAD/CAM equipment *Journal of Esthetic Dentistry* **11(4)** 206-214.

Sulaiman F, Chai J, Jameson LM & Wozniak WT (1997) A comparison of the marginal fit of In-Ceram, IPS Empress, and Procera crowns *International Journal of Prosthodontics* **10(5)** 478-484.

Thordrup M, Isidor F & Hörsted-Bindslev P (1999) A 3-year study of inlays milled from machinable ceramic blocks representing 2 different inlay systems *Quintessence International* **30(12)** 829-836.

Van Meerbeek B, Inokoshi S, Willems G, Noack MJ, Braem M, Lambrechts P, Roulet JF & Vanherle G (1992) Marginal adaptation of four tooth-colored inlay systems *in vivo Journal of Dentistry* **20(1)** 18-26.

©Operative Dentistry, 2004, **29-2,** 219-225

In Situ and *In Vitro* Effects of Bleaching with Carbamide Peroxide on Human Enamel

LM Justino • DR Tames • FF Demarco

Clinical Relevance

The adverse effects of vital bleaching with carbamide peroxide were not observed when simulating the oral condition. Saliva could have a remineralizing effect over bleached enamel.

SUMMARY

This study evaluated *in vitro* and *in situ* the potential adverse effects of 10% carbamide peroxide on human enamel using microhardness, calcium loss and surface morphology analysis. Twenty-four enamel slices (4 mm²) were obtained from recently extracted premolars. The specimens were polished under water-cooling down to 1,200-grade sandpaper. After initial microhardness readings (100g), the specimens were randomly divided into two groups for *in situ* and *in vitro* conditions. The specimens were covered with 10% carbamide peroxide for eight hours. After removing the bleaching gel, the *in vitro* specimens were stored in deionized water and the *in situ* specimens, included in an intra-oral appliance, were placed in the oral cavity of four volunteers. These cycling sequences took place for 14 days. Upon conclusion of the bleaching treatment, new microhardness readings were performed on all specimens. Calcium dosage was assessed from the bleaching gel collected after initial exposure on day one, then from gel collected between days two and seven and gel collected between day eight and 14 using an atomic absorption spectrophotometer. Surface morphology was observed from two non-treated control specimens and two specimens of each experimental bleached group under SEM evaluation. Statistical analysis (ANOVA and Tukey tests) disclosed that specimens bleached *in situ* showed similar microhardness to unbleached specimens and had statistically higher ($p<0.01$) hardness than *in vitro* bleached specimens. The loss of calcium in the *in vitro* situation at 14 days was 2.5 times higher than the *in situ* condition. SEM micrographs demonstrated that surface alterations were more pronounced in the *in vitro* condition. The adverse effects of carbamide peroxide on enamel were evident in specimens bleached *in vitro* but were not seen *in situ*. The presence of saliva could prevent the demineralizing effect of bleaching gel *in situ*.

Lídia Morales Justino, DDS, MS, assistant professor, Universidade do Vale do Itajaí, Itajaí, SC, Brasil

David Rivero Tames, DDS, MS, PhD, associate professor, Universidade do Vale do Itajaí, Itajaí, SC, Brasil

*Flávio Fernando Demarco, DDS, PhD, professor, Department of Operativy Dentistry, Federal University of Pelotas, Pelotas-RS, Brasil

*Reprint request: Rua Gonçalves Chaves, 457—Centro, Pelotas-RS, Brasil, CEP 96015-560; e-mail: sancha@uol.com.br

INTRODUCTION

The demands of the population regarding their dental appearance are greater than ever today. Teeth need to be not only aligned properly, they must also be white (Goldstein & Garber, 1995). This, along with a decrease

in dental caries, has directed clinicians to conservative aesthetic dentistry with non-restorative treatments for discolored teeth, an area under rapid development. This has led to the widespread use of bleaching agents to lighten darkened teeth. The technique of bleaching vital teeth is easy and patient acceptance is high.

The nightguard vital bleaching technique, with a build-in reservoir, offers a conservative, cost effective method for bleaching teeth (Haywood & Heymann, 1989). Most commercial products contain 10% carbamide peroxide with carboxypolymethylene polymer (carbopol) as a thickening agent; this polymer improves tissue adherence and allows for longer time release of the bleaching agent (Haywood, 1992; Rodrigues & others, 2001). Despite the favorable results achieved with nightguard vital bleaching, some reports in the literature have related adverse side effects as a consequence of the treatment. Sensitivity following the treatment has been related to the possible removal of mineral content from enamel and dentin (Bitter, 1998). Also, a reduction in enamel bond strength of the adhesive materials has been linked to physical and chemical alterations caused by bleaching materials (Ben-Amar & others, 1995).

There are a number of methods that have been used to assess the effects of bleaching agents on enamel. They involve *in vitro* and *in vivo* exposure to the bleaching agent and the subsequent assessment by visual inspection, SEM, hardness testing and surface loss.

McCraken and Haywood (1995) found an alteration in surface hardness and calcium loss after enamel treatment with 10% carbamide peroxide. A severe reduction in enamel hardness and distinct alterations in the surface morphology of bleached teeth were also reported (Ben-Amar & others, 1995; Josey & others, 1996; Smidt & others, 1998). In a SEM study, Tames, Grando and Tames (1995) found significant surface alterations in enamel topography following enamel bleaching with 10% carbamide peroxide. Different bleaching materials could produce an increase or decrease in enamel microhardness due to bleaching time (Rodrigues & others, 2001). Despite detecting a decrease in the microhardness of dentin bleached with 10% carbamide peroxide, de Freitas and others (2002) have verified that 14 days post-treatment, microhardness values were recovered to baseline levels. Performing a histochemical analysis of dental hard tissues following bleaching, Rotstein and others (1996) detected a significant reduction in Ca/P ratio. These authors advised that bleaching materials could adversely affect dental hard tissues and should be used with caution. An *in vitro* study by Flaitz and Hicks (1996) showed that different concentrations of carbamide peroxide can remove mineral structures from enamel, causing morphological alterations with different forms and intensity and can reach to the subsurface. Crews and others (1997) verified that different bleaching agents have been known to lower the Ca and P levels in human enamel. Cimilli and Pameijer (2001) showed that some bleaching formulations could lower enamel hardness and cause the dissolution of calcium. In contrast, Murchison, Charlton and Moore (1992) noticed no significant changes in hardness of human enamel *in vitro* after exposure to 10% carbamide peroxide. Lee and others (1995) have also detected no influence on microhardness in bleached enamel using 10% carbamide peroxide *in vitro* condition but found an evident alteration to the enamel surface under SEM examination.

Despite the differences found *in vitro* studies, the clinical significance of these alterations and whether or not they have relevance in daily practice remains unknown (McCraken & Haywood, 1996; Cimilli & Pameijer, 2001). Nevertheless, it has been proposed that the loss of mineral content and increased porosity could explain transitory dental sensitivity during bleaching treatment (Basting, Rodrigues & Serra, 2001).

The *in vivo* dynamic interaction of saliva/enamel is a factor that has generally not been incorporated into *in vitro* experiments (Cimilli & Pameijer, 2001). Saliva has a cleaning action, a buffering capacity and a remineralization ability (Cury, 1989; Thylstrup & Fejerskov, 1998) that could prevent the adverse demineralizing effect of the bleaching agent. In an *in vivo* study, Shannon and others (1993) found lower hardness values for bleached enamel but without statistical significance. In a recent *in situ* study, Basting and others (2001) verified that 10% carbamide peroxide agent altered the microhardness of sound, demineralized enamel but not the microhardness of sound, demineralized dentin. Using an *in vivo* model, Bitter (1998) bleached teeth indicated for extraction and evaluated them for surface morphology using SEM. Non-bleached teeth were used as the control. This author verified surface modifications even after 90 days, suggesting that such alterations could be responsible for sensitivity.

Since little information exists in the literature regarding the clinical response to bleaching treatment, there is a need for studies that simulate clinical conditions in order to evaluate the real effects of such treatment. The hypothesis to be tested is that in a clinical oral simulate condition (*in situ*), the effects of bleaching agents are less evident than when seen in *in vitro* conditions. This study investigated the effect of 10% carbamide peroxide on human enamel using *in situ* and *in vitro* methodologies.

METHODS AND MATERIALS

Preparation of the Enamel Slabs

Twelve recently extracted maxillary premolars, removed for orthodontic reasons, were used in this study. The teeth were selected from six individuals between 12 and 14 years of age. All teeth were exam-

ined under magnification (20x) to detect micro-cracks and surface defects. Only premolars without defects were selected for this study. Soon after extraction, the crowns were cut at the CE-junction using a diamond saw under copious water-cooling. The pulp tissue was removed and the teeth were freezer stored. To perform the tests, the crowns were sectioned longitudinally to obtain 4 mm^2 enamel slabs. The enamel slabs were subjected to steam sterilization to avoid bacterial contamination (Amaechi, Higham & Edgar, 1998).

Each slab was included in a PVC matrix using polystyrene matrix, keeping only one side unsealed. Samples were sequentially polished by means 400, 600 and 1200 grade sandpaper. Twenty-four enamel slabs were obtained. Two slabs were used as a control for SEM evaluation and remained unbleached. The enamel slabs were randomly divided into two bleaching groups (n=11), one performed *in situ* and the other *in vitro*.

Exposure of the Slabs to Bleaching Agent (*in situ* condition—experimental condition)

A commercial bleaching agent Whiteness (FGM, Joinville, SC, Brazil) was used. This gel is a 10% carbamine peroxide agent with carboxypolymethylene polymer (carbopol), having a pH=7.82.

Four undergraduate dental students from the Federal University of Pelotas were used as volunteers for the bleaching treatments. All the procedures were carried out upon approval from the Ethic Committee, and the students provided written consent to partake in the study. Impressions were taken and removable oral appliances were made for each individual. Eleven enamel slabs were removed from individual matrixes using probes and were included in the oral appliances: three volunteers received three enamel slabs and the remaining volunteer received two. The bleaching procedure was performed outside the oral cavity. The enamel slabs were covered with 0.05 ml of 10% carbamine peroxide gel for eight hours at night, with the oral appliance being covered with a PVC film. The bleaching gel was then removed and the oral appliances placed in the mouths of the four volunteers for 16 hours to simulate clinical conditions and the effects of saliva on bleached enamel.

Exposure of the Slabs to Bleaching Agent (*in vitro* condition—control condition)

Each enamel slab in the individual matrix received an application of 0.05 ml of 10% carbamide peroxide gel for eight hours. After removing the gel, the enamel slabs were placed in individual containers with deionized water for 16 hours. Both bleaching treatments were performed for 14 days.

Microhardness Test

Microhardness measurements were performed before both bleaching treatments. Vickers microhardness was measured using a microhardness tester (Buehler, Model 1600, Lake Buff, IL, USA). Three indentations were made on each specimen with 100g for five seconds. The means of each specimen was transformed in Vickers hardness number (VHN) based on a specified calculation formula.

After 14 days of *in situ* or *in vitro* bleaching treatments, the enamel slabs were again tested for microhardness.

Calcium Loss Evaluation

The bleaching gel from the *in vitro* or *in situ* conditions was collected daily. Three measurements of the calcium content were made for each condition: on the first day, on the seventh day from the gel collected from the second to the seventh day and at 14^{th} day using the gel collected between the eighth and 14^{th} day. The gel removed after bleaching treatment at each period was added to 0.5 ml of deionized water, resulting in the 10% initial dilution necessary for determining calcium loss. Samples were stored at 8°C.

The calcium readings were performed using an Atomic Absorption Spectophotometer with a detection limit for Ca of 0.05 µg. The results were expressed in µg Ca/ml gel.

Surface Morphology Analysis

Six enamel slabs were separated for SEM analysis: two non-bleached specimens, two specimens from the *in situ* condition and two from the *in vitro* condition. The specimens were vacuum desiccated in alcohol and acetone and submitted to gold sputtering (500 A). Micrographs were taken at different magnifications (600x and 2000x) using a Phillips XL 20 microscope (15 KVa). Figure 1 provides a graphic representation of the methods and materials.

RESULTS

Microhardness Tests

Since data from the microhardness evaluation were normal, parametric tests were employed. Two factors were investigated: the condition (*in situ* x *in vitro*) and the time (before and after bleaching treatment).

The ANOVA test disclosed differences between *in situ* and *in vitro* conditions and between pre- and post-bleaching specimens. Also, interaction between the condition and time was significant, demonstrating that specimens showed a different performance for the same condition.

An additional Tukey post-hoc test was used to clarify differences among the groups. In Table 1, means for the different conditions and times are expressed and the Tukey interval is presented.

In Table 1, it can be noted that the initial specimens from the *in situ* and *in vitro* conditions exhibited similar

hardness values. The hardness of the *in situ* specimens after bleaching treatment was similar to that found in the initial condition. However, bleached specimens from the *in vitro* condition had the lowest hardness values, which was statistically different from all others groups ($p<0.05$).

Calcium Loss Evaluation

Table 2 shows the results of the amount of calcium found in gel removed at different times. Analysis of the gel demonstrated that greatest removal occurred during the first day under both conditions. Calcium removal decreased drastically during the second period (second to seventh day) and showed a further small reduction in the third period evaluated (days eight to 14). These findings suggest that the bleaching agent first attacks the superficial layer of the crystallites. The loss of calcium was always significantly higher *in vitro* than *in situ*. The total amount of calcium lost during the entire experiment was 2.5 times higher *in vitro* than *in situ*.

Surface Morphology Analysis

SEM analysis of the surface showed different patterns in the specimens evaluated. Figure 1 shows the appearance of the enamel surface from unbleached specimens (2000x). The uniform enamel pattern observed, including tiny depressions and elevations, is due to polishing procedures. The surface observed in specimens bleached *in situ* shows patterns similar to the unbleached condition; however, the depressions observed *in situ* are more evident than those in the control slabs (Figure 2). More evident depressions are observed *in vitro* (Figure 3), which indicates a higher mineral loss due to dissolution of the enamel rods. In some regions, bleached enamel resembles phosphoric acid etched enamel.

DISCUSSION

In this study, microhardness, calcium removal and surface analysis were investigated after enamel treatment with 10% carbamide peroxide with carbopol using *in situ* and *in vitro* methodologies.

A significant effect of bleaching was noted in the enamel specimens in the *in vitro* situation. This was combined with a significant decrease in microhardness, higher calcium loss and deep surface alterations. Such findings confirm the previous results that were reported in the literature that relates to the detrimental demineralizing *in vitro* effect of bleaching agents on enamel (Ben-Amar & others, 1995; McCracken & Haywood, 1995; Tames, Grando & Tames, 1995; Josey & others, 1996; Flaitz & Hicks, 1996; Rotstein & others, 1996; Crews & others, 1997; Smidt & others, 1998; Cimilli & Pameijer, 2001; Rodrigues & others, 2001).

In contrast, when the results of the *in situ* condition were observed, no significant differences were seen. In relation to surface hardness, the values were similar to unbleached enamel. Morphology analysis disclosed a surface similar to normal enamel, with few alterations from the control group. Also, the amount of calcium loss was significantly lower in *in situ* specimens than for *in vitro* specimens. Using similar methodology to evaluate the microhardness after 10% carbamide peroxide treatment on enamel, Shannon and others (1993) found similar results. However, the findings from this study are not in accordance with those found *in situ* by Basting and others (2001), who found enamel and dentin fragments, sound or demineralized, were bonded in the buc-

Table 1: *Means (SD) of Microhardness (VHK) for Different Conditions*

	In Situ	*In Vitro*
Initial	333.88 (± 19.59)[a]	341.68 (± 14.95)[a]
Post bleaching	321.66 (± 30.78)[a]	206.00 (± 40.79)[b]
Tukey interval (5%)=27.99		

[a]*Mean values with the same subscript letters were not statistically different (p>0.05).*

Table 2: *Calcium Content (mg/ml) Present in Gel Removed at the Different Periods Evaluated*

	In Situ	*In Vitro*
1st day	586.63	1,210.36
2nd to 7th day	18.00	250.85
8th to 14th day	7.82	55.14
Total amount	611.45	1,516.35

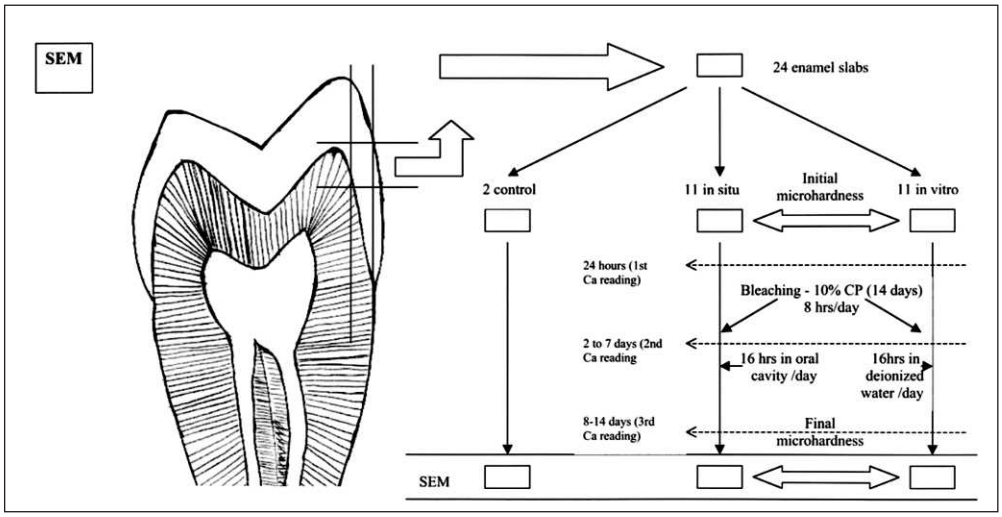


Figure 1. *Illustration of the methodology used in the study.*

cal surface of upper first molars or second premolars. Their volunteers were submitted to one double blind study using 10% carbamide peroxide or a placebo agent. They also detected a significant decrease in microhardness in both sound or mineralized enamel despite the presence of saliva, fluorides and plaque control. Nevertheless, no significant decrease in dentin microhardness was found.

Differences in the methodologies employed could influence the results found. In the current study, enamel fragments were placed in oral appliances situated in the palate region and the bleaching treatment was performed outside the oral cavity. In a study by Basting and others (2001), the enamel slabs were bonded to the upper molars and premolars, with all the procedures performed in the oral cavity. Also, despite both studies using 10% carbamide peroxide, the materials present with differences in composition such as pH or the amount of carbopol. The pH of the Opalescence material is 6.68 (Basting & others, 2001), while the bleaching agent used in the current study (Whiteness) has a pH=7.82. The most pronounced changes were found in the enamel slabs exposed to lower-pH solutions (Shannon & others, 1993). Bleaching treatments for the two studies were also different, three and two weeks, respectively. More severe changes have been observed in prolonged bleaching treatments (Shannon & others, 1993) and different bleaching times can produce an increase or decrease in enamel hardness (Rodrigues & others, 2001).

In this study, differences in the *in situ* and *in vitro* condition could be attributed to the important role of human saliva in the remineralization process. While enamel slabs from the *in vitro* methodology were stored in deionized water, slabs from the *in situ* group were submitted to a clinical condition in oral appliances used by four volunteers. The oral environment provides conditions for enamel remineralization and demineralized enamel is more susceptible to remineralization (Ten Cate, 1990). When the bleaching agent causes demineralization in enamel, ionic changes are induced, increasing mineral uptake, which replaces the mineral lost during treatment. For example, deciduous enamel eroded by lemon juice was found to be more reactive than non-eroded enamel, and a higher fluoride deposition was observed in eroded enamel after applying 2% neutral sodium fluoride (Rath, 1995).

Saliva has a cleaning function, but it also has a buffering action due to the bicarbonate and phosphate systems. Some inorganic electrolytes contained in saliva (calcium, phosphor and fluorides) are important participants in the remineralization process. When pH is under the physiologic limit, part of the calcium and phosphor complexes are released and added to the ionic

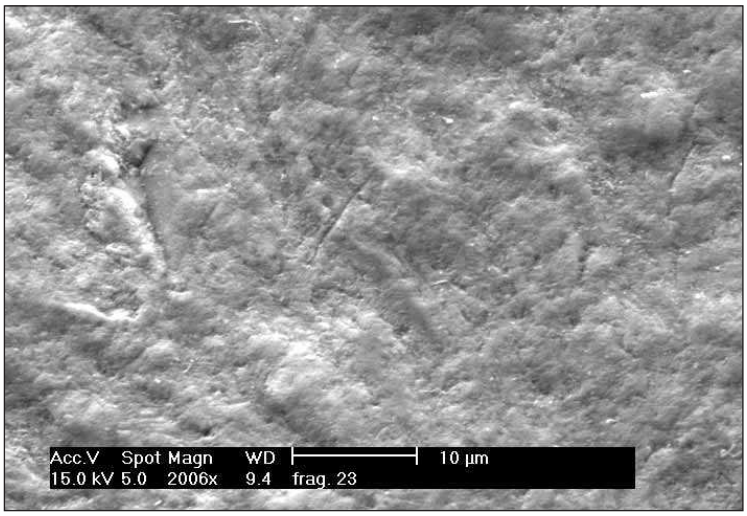


Figure 2. *Enamel slab from control group that was not submitted to any bleaching treatment. The tiny depressions and elevations are related to the polishing procedures (2006x).*

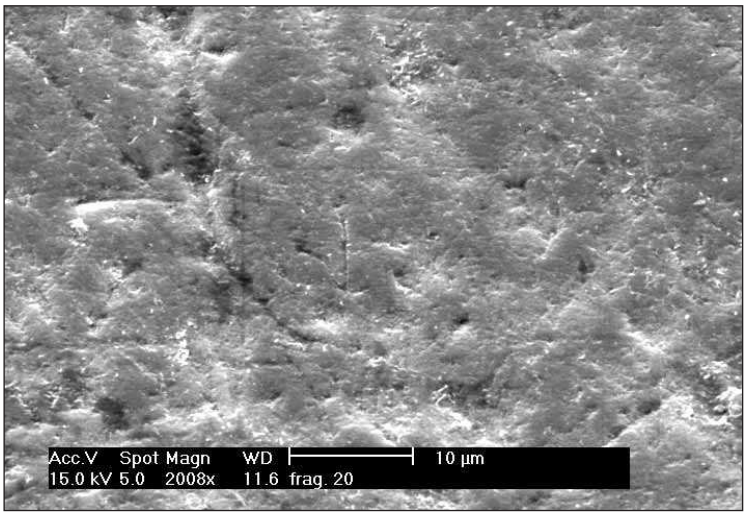


Figure 3. *Enamel slab bleached* in situ *disclosing similar pattern to the unbleached specimens, but the depressions observed are more evident (2008x).*

calcium and phosphor reservoirs. Consequently, apatite from the enamel surface is protected against dissolution (Thylstrup & Fejerskov, 1998).

There is a correlation between the values of microhardness and calcium loss evaluations allowed by the dynamics of the oral cavity. In a pH of less than 5.5, the amount of Ca and P in saliva is lower than the solubility rate of hydroxyapatite, with enamel having a tendency to lose Ca and P to the oral environment (Cury, 1989). In the enamel slabs cycled *in situ*, saliva interfered in this process, allowing the reposition of mineral and the reestablishment of hardness values similar to non-bleached specimens. However, in enamel slabs cycled *in vitro*, there was no remineralization effect because of the absence of saliva.

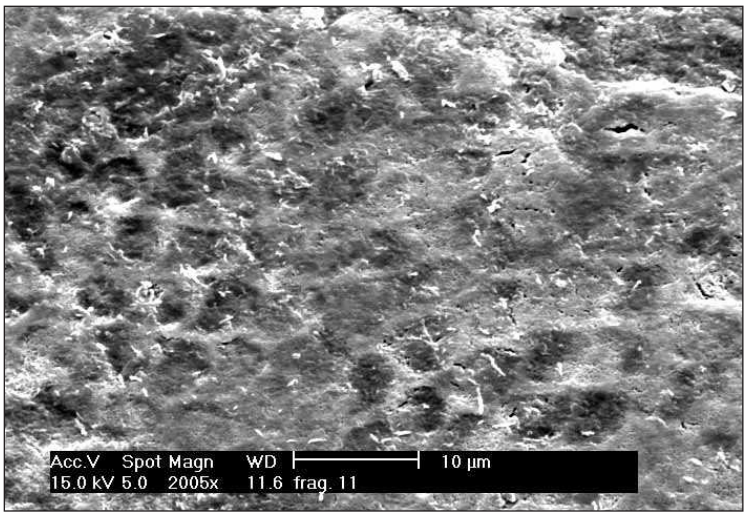


Figure 4. *Pronounced depressions are observed in the enamel slab bleached* in vitro. *The higher mineral loss probably is due to the dissolution of enamel rods. A surface that resembles acid etched enamel is observed in some regions of the* in vitro *bleached slab (2005x).*

The pH and viscosity of bleaching agents could influence the degree of demineralization on bleached enamel (Smidt & others, 1998). The acidic properties of bleaching agents, prolonged contact time between the bleaching agent and dental structure and the presence of a high percentage of carbopol have been indicated as potential factors responsible for surface alterations (Basting & others, 2001). However, a moderately low-pH bleaching agent solution *in vivo* reduces the pH of saliva in the mouth during the first five minutes. Over the next 15 minutes of treatment, the pH increased above baseline due to the chemical reactions of carbamide peroxide and the neutralizing effect of saliva (Leonard, Bentley & Haywood, 1994). Since the gel in this study presents a non-acidic pH (7.82), changes observed in the enamel structure could be due to the prolonged contact time with the bleaching agent or carbopol concentration.

Although enamel microhardness decreased and the surface morphology suffered a significant change after treatment with carbamide peroxide in a laboratory study, Smidt and others (1998) stated that the buffering capacity and the remineralization potential of saliva might overcome detrimental bleaching effects *in vivo*. Even when artificial saliva is used to simulate the natural saliva function, the detrimental effects of bleaching were less evident (Rodrigues & others, 2001; de Freitas & others, 2002).

Cimilli and Pameijer (2001) pointed out that it is difficult to determine the clinical significance of the results from *in vitro* studies since the calcium and phosphates available in saliva can potentially replenish the lost substance. Some authors have also highlighted the limitations of *in vitro* studies to determine the effects of bleaching on enamel (Leonard, Bentley & Haywood, 1994; Shannon & others, 1998; Smidt & others, 1998; Rodrigues & others, 2001). These limitations reinforce the relevance of this study. The similarity of clinical conditions provided by the *in situ* methodology can offer new perspectives in evaluation of the effects of different dental treatments.

Data from *in situ* and *in vitro* methodologies confirm the correlation between the factors evaluated in this study. When there is significant mineral loss, microhardness values tend to decrease and surface alterations are evident. However, when mineral removal is lower and is replaced with mineral from saliva, the hardness tends to be similar to the control group and surface alterations are less evident.

The results of this study confirmed the hypothesis tested and showed that in simulated clinical conditions bleaching does not produce the detrimental effects observed on enamel *in vitro*. This supports the reported clinical experience that 10% carbamide peroxide vital bleaching is a safe procedure that can be performed in daily clinical practice (Ritter & others, 2002).

CONCLUSIONS

The limitations of this study show that the remineralization effect of saliva could prevent the demineralization effect of bleaching treatment in human enamel *in situ*.

Acknowledgements

The authors thank Dr Richard Van Noort, University of Sheffield, for his help and suggestions during the preparation of this manuscript.

(Received 10 March 2003)

References

Amaechi BT, Higham SM & Edgar WM (1998) Efficacy of sterilization methods and their effect on enamel demineralization *Caries Research* **32(6)** 441-446.

Basting RT, Rodrigues AL Jr & Serra MC (2001) The effect of 10% carbamide peroxide bleaching material on microhardness of sound and demineralized enamel and dentin *in situ Operative Dentistry* **26(6)** 531-539.

Ben-Amar A, Liberman R, Gorfil C & Bernstein Y (1995) Effect of mouthguard bleaching on enamel surface *American Journal of Dentistry* **8(1)** 29-32.

Bitter NC (1998) A scanning electron microscope study of the long-term effect of bleaching agents on the enamel surface *in vivo General Dentistry* **46(1)** 84-88.

Cimilli H & Pameijer CH (2001) Effect of carbamide peroxide bleaching agents on the physical properties and chemical composition of enamel *American Journal of Dentistry* **14(2)** 63-66.

Crews KM, Duncan D, Lentz D, Gordy FM & Tolbert B (1997) Effect of bleaching agents on chemical composition of enamel *Mississippi Dental Association Journal* **53(1)** 20-21.

Cury JA (1989) [Uso de flúor (Fluoride usage)] in: Baratieri LN, Monteiro S Jr, Caldeira MA & others [Dentística—Procedimentos preventives e restauradores] *Operative Dentistry—Preventive and Restorative Procedures* Rio de Janeiro Santos 43-68.

de Freitas PM, Basting RT, Rodrigues JA & Serra MC (2002) Effects of two 10% peroxide carbamide bleaching agents on dentin microhardness at different time intervals *Quintessence International* **33(5)** 370-375.

Flaitz CM & Hicks MJ (1996) Effects of carbamide peroxide whitening agents on enamel surfaces and caries-like lesion formation: A SEM and polarized light microscopic *in vitro* study *ASDC Journal of Dentistry for Children* **63(4)** 249-256.

Goldstein RE & Garber DA (1995) *Complete Dental Bleaching* Chicago Quintessence p 165.

Haywood VB & Heymann HO (1989) Nightguard vital bleaching *Quintessence International* **20(3)** 173-176.

Haywood VB (1992) History, safety and effectiveness of current bleaching techniques and applications of the Nightguard vital bleaching technique *Quintessence International* **23(7)** 471-488.

Josey AL, Meyers IA, Romaniuk K & Symons AL (1996) The effect of a vital bleaching technique on enamel surface morphology and the bonding of composite resin to enamel *Journal of Oral Rehabilitation* **23(4)** 244-250.

Lee CQ, Cobb CM, Zargartalebi F & Hu N (1995) Effect of bleaching on microhardness, morphology and color of enamel *General Dentistry* **43(2)** 158-162.

Leonard RH Jr, Bentley CD & Haywood VB (1994) Salivary pH changes during 10% carbamide peroxide bleaching *Quintessence International* **25(8)** 547-550.

McCracken MS & Haywood VB (1995) Effects of 10% carbamide peroxide on the subsurface hardness of enamel *Quintessence International* **26(1)** 21-24.

McCracken MS & Haywood VB (1996) Demineralization effects of 10% carbamide peroxide *Journal of Dentistry* **24(6)** 395-398.

Murchison DF, Charlton DG & Moore BK (1992) Carbamide peroxide bleaching: Effects on enamel surface hardness and bonding *Operative Dentistry* **17(5)** 181-185.

Rath IBS (1995) [Análise morfológica e bioquímica da deposição de fluoretos no esmalte de dente decíduo humano erosionado pelo suco de limão. Estudo *in vitro*] Morphologic and biochemistry analysis of the deciduous human enamel eroded by lemmon juice. *In vitro* study (Master of Sciences Thesis) Federal University of Santa Catarina p 135.

Ritter AV, Leonard RH Jr, St. Georges AJ, Caplan DJ & Haywood VB (2002) Safety and stability of nightguard vital bleaching: 9 to 12 years post-treatment *Journal of Esthetic and Restorative Dentistry* **14(5)** 275-285.

Rodrigues JA, Basting RT, Serra MC & Rodrigues AL Jr (2001) Effects of 10% carbamide peroxide bleaching on enamel microhardness *American Journal of Dentistry* **14(2)** 67-71.

Rotstein I, Dankner E, Goldman A, Heling I, Stabholz A & Zalkind M (1996) Histochemical analysis of dental hard tissues following bleaching *Journal of Endodontics* **22(1)** 23-25.

Shannon H, Spencer P, Gross K & Tira D (1993) Characterization of enamel exposed to 10% carbamide peroxide bleaching agents *Quintessence International* **24(1)** 39-44.

Smidt A, Weller D, Roman I & Gedalia I (1998) Effect of bleaching agents on microhardness and surface morphology of tooth enamel *American Journal of Dentistry* **11(2)** 83-85.

Tames D, Grando LJ & Tames DR (1995) [Alterações do esmalte dental submetido ao tratamento com peróxido de carbamida a 10%] Enamel alterations following treatment with 10% carbamide peroxide *Revista da Associação Paulista de Cirurgiões Dentistas* **52(1)** 145-149.

Ten Cate JM (1990) *In vitro* studies on the effects of fluoride on de- and re-mineralization *Journal of Dental Research* **69(Special Issue)** 614-619.

Thylstrup A & Fejerskov O (1998) [Tratado de cariologia] [Cariology] Rio de Janeiro, *Cultura Médica* p 388.

©Operative Dentistry, 2004, **29-2,** 226-233

Literature Review

Reattachment of Fractured Teeth: A Review of Literature Regarding Techniques and Materials

A Reis • AD Loguercio
A Kraul • E Matson

Clinical Relevance

Regarding the materials used for reattachment, evidence-based literature reviews show that materials do not play an important role in fracture strength recovery. The combination of light-cured, single-bottle adhesives and chemical-cured luting cements or chemical-cured composites may be avoided due to an incompatibility among components.

Regarding techniques, it is not advisable to employ simple reattachment without additional preparation since its fracture strength recovery is approximately 40% of intact teeth. Placement of an internal groove is an excellent alternative when the remnant and fragment fit well, while an overcountourn should be used in cases where the loss of structure occurred in a fractured site.

SUMMARY

Anterior crown fractures are a common form of injury that mainly affects children and adolescents. The position of maxillary incisors and their eruptive pattern carries a significant risk for trauma. In the pre-adhesive era, fractured teeth needed to be restored either with pin-retained inlays or cast restorations that sacrificed healthy tooth structure and were a challenge for clinicians to match with adjacent teeth. The development of adhesive dentistry has allowed dentists to use the patient's own fragment to restore the fractured tooth. Since then, several successful case reports that use a variety of techniques and materials to reattach fractured teeth have been published. This article presents a comprehensive literature review on techniques and materials used to restore uncomplicated dental trauma.

*Alessandra Reis, DDS, PhD, professor, University of Oeste de Santa Catarina, UNOESC–Joaçaba, SC, Brazil, Department of Dental Materials and Restorative Dentistry, Joaçaba, SC, Brazil

Alessandro Dourado Loguercio, DDS, MS, PhD, professor, University of Oeste de Santa Catarina. UNOESC, Joaçaba, SC, Brazil, Department of Dental Materials and Restorative Dentistry, Joaçaba, SC, Brazil

Alexander Kraul, DDS, MS, graduate student, Department of Operative Dentistry, School of Dentistry, University of São Paulo, USP, São Paulo, SP, Brazil

Edmir Matson, DDS, MS, PhD, professor and chairman, Department of Operative Dentistry at University of São Paulo, USP, São Paulo, SP, Brazil

*Reprint request: Rua Getúlio Vargas, 2125, Bairro Flor da Serra, CEP 89600-000, Joaçaba, SC, Brasil; e-mail: reis_ale@hotmail.com

INTRODUCTION

Recent published studies on the incidence of dental trauma indicate that there is an increase in the number of these lesions among children and teenagers. Different classification systems of traumatic tooth fractures appear in the literature (Andreasen & Andreasen, 1993; Ellis & Davey, 1970; Spinas & Altana, 2002; Baratieri, Monteiro & Andrada, 1998). Tables 1, 2, 3

Table 1: *Andreasen and Andreasen's Classification (1993)*

Class I	Enamel infraction (crack)
Class II	Enamel fracture (crown fracture, not complicated)
Class III	Enamel-dentin fracture (crown fracture, not complicated)
Class IV	Complicated crown fracture
Class V	Crown-root fracture, not complicated
Class VI	Complicated crown-root fracture
Class VII	Root fracture

Table 2: *Ellis and Davey's Classification (1970)*

Class I	Simple crowns fracture with plain enamel involvement
Class II	Extended crown fracture with noticeable dentinal involvement, without pulp exposition
Class III	Extended crown fracture with noticeable dentinal involvement, with pulp exposition
Class IV	Teeth that lost their vitality, with or without loss of crown tissues
Class V	Traumatically avulsed teeth
Class VI	Crown fracture, with or without loss of crown tissues
Class VII	Tooth luxation withour crown or root fracture
Class VIII	Cervical crown fracture
Class IX	Traumatic injuries on primary dentition

Table 3: *Spinas and Altana's Classification (2002)*

A Class	Simple enamel lesions involving one proximal angle or only incisal edge.
B Class	Enamel-dentin lesions involving one proximal angle or only an incisal edge.
	Subclass b1 – with pulp exposition
C Class	Enamel-dentin lesions involving the incisal edge and at least a third of the crown.
	Subclass c1 – with pulp exposition
D Class	Enamel-dentin lesions involving the mesial or distal angle and the incisal or palatal surface and root involvement.
	Subclass d1 – with pulp exposition

*****When necrotic pulp is present, the letter "h" is placed after the main class.*

Table 4: *Baratieri, Monteiro and Andrada's Classification (1998)*

A Class		**Enamel Fracture**
		B1 - Without pulpal and biologic width involvement
B Class	Enamel-dentin Fracture	*B2* - Without pulpal involvement but biologic width violation (violation at or coronal to the bone crest level)
		B3 - With pulpal involvement and no biologic width violation
		B4 - With pulpal involvement and biologic width violation (violation at or coronal to the bone crest level)

and 4 show these classification systems. In spite of differences in the percentage rates and classification systems used, a majority of the studies agree in several respects: 1) the most common injuries are uncomplicated crown fractures (Ellis Class I and II; Andreasen Class I, II and III, which represent enamel and enamel-dentin fractures without pulp exposure); 2) children and teenagers are most affected, with boys being the highest risk group; 3) upper central incisors are most affected and 4) traffic accidents and "at risk" athletic activities are usually the most common causes of dental trauma (Zerman & Cavalleri, 1993; Çaliskan & Türkün, 1995; Cavalleri & Zerman; 1995; Hamilton, Hill & Holloway, 1997; Murchison, Burke & Worthington, 1999; Dietschi & others, 2000).

The incidence of dental trauma is on the rise due to an increase in dangerous activities and sports that involve children (Zerman & Cavalleri, 1993). Fragment reattachment is the preferred technique among clinicians because it has several advantages over conventional acid-etch composite restorations. Improved esthetics is obtained since enamel's original shape and color, brightness and surface texture are maintained. In addition, the incisal edge wears at a similar rate to adjacent teeth, whereas, a composite restoration will likely wear more rapidly. Furthermore, this technique is less time consuming and provides more predictable long-term wear (Baratieri, Monteiro & Andrada, 1990).

Although unsupported by laboratory studies or clinical trials, several successful case reports related to fragment reattachments were published in the mid-to-late 1980s. An assortment of techniques and materials were employed with no consensus regarding their advantages and disadvantages.

Therefore, this article reviews literature on the techniques and materials used and emphasizes the advantages and disadvantages of each. Discussion is restricted to simple coronal fractures without periodontal and pulp involvement (shaded rows in Tables 1, 2, 3 and 4), since these more complicated injuries make up only a small percentage of the total injuries of permanent dentition (Andreasen, Daugaard-Jensen & Munksgaard, 1991).

BACKGROUND

Innovative techniques to treat fractured teeth were only recently documented in the literature due to the difficulty of fragment retention to the remaining crown. Chosack and Eildeman published the first case report on reattachment of a fractured incisor fragment in 1964. The authors managed a complicated case where, after

endodontic treatment, a cast post and core were cemented. The post and core were fitted to the prepared tooth fragment and attached to the remnant using an unspecified cement. The authors termed this treatment as a temporary restoration. In 1977, Spasser performed the endodontic treatment and the tooth fragment was retained with three dentin pins.

In the late 1970s, Tennery (1978), Starkey (1979) and Simonsen (1979) reported cases of fragment reattachment using enamel etching and resin composites The first follow-up examination (two years and six months later) was overwhelmingly positive in terms of retention (Esberard, Silva Filho & Gabrielli, 1978).

Since then, a series of other case reports on fragment reattachment have been published despite being unsupported by laboratory studies or clinical trials (Murchison & Worthington, 1998). Clinicians employed an assortment of beveled designs, chamfers, endodontic adjunctive care, dentinal and enamel grooves and choices of resin composite materials and techniques.

LITERATURE DISCUSSION

Reattachment Techniques

Enamel Beveling

Several case reports advocate *enamel beveling* of the fragment and the remaining crown as shown in Figure 1A (Simonsen, 1979; Amir, Bar-Gil & Sarnat, 1986; Burke, 1991; Van Der Vyner & Marais, 1996; Walker, 1996). In some case reports, this technique is performed only on the lingual surface (Simonsen, 1982) instead of the whole fractured area (circumferential bevel). This technique has claimed to improve fragment retention since enamel beveling alters the enamel prisms orientation, allowing for achievement of a more effective acid etching pattern (Simonsen, 1979; Bagheri & Denehy, 1983). This technique also improves short-term esthetics (Fahl, Denehy & Jackson, 1995; Fahl, 1997).

V-shaped Internal Enamel Groove

Another technique commonly reported in the literature is placement of a *V-shaped internal enamel groove* (Figure 1B) (Simonsen, 1982; Diangelis & Jungbluth, 1992) which is then restored with resin composite. Clinically, this technique is difficult to perform due to the limited enamel thickness of anterior teeth.

However, instead of placing a bevel in enamel, this procedure can be performed in dentin. Actually, placing an internal groove in dentin from the fragment and the remaining tooth has been done in order to create space for a pulp-capping agent (Simonsen, 1982; Franco & others, 1985; Baratieri & others, 1990) or for adhesive cement-like glass ionomers (Diangelis & Jungbluth, 1992; Burke, 1991).

Internal Dentin Groove

However, the space provided by the *internal dentin groove* (Figure 1C) has since been utilized as reattachment reinforcement with resin composites (Walker, 1996; Reis & others, 2001). Similarly, this kind of reinforcement has already been performed in pulpless teeth, where part of the pulp chamber was filled with resin composite (Amir & others, 1986; Diangelis & Jungbluth, 1992). Some authors claim that this technique compromises esthetics, as the internal resin composite can modify the shade of teeth (Baratieri & others, 1998). However, one may consider this as groove-size and material dependent.

External Chamfer

The previous techniques present a common drawback. Since they require enamel and/or dentin preparation

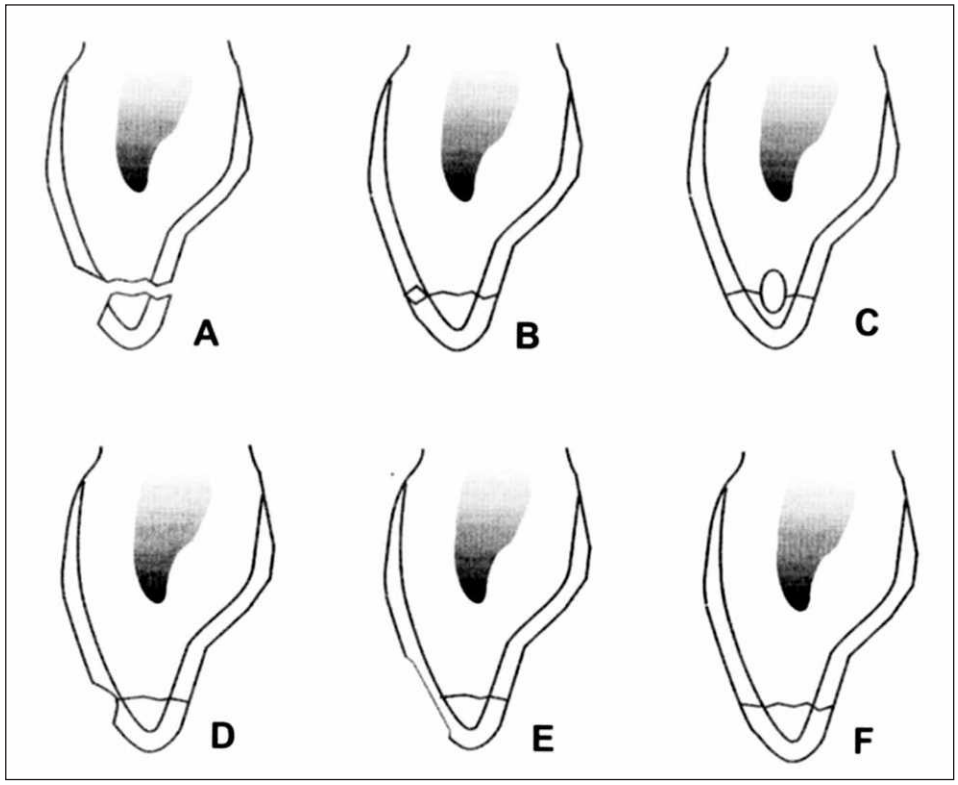


Figure 1. *The most common reattachment techniques for uncomplicated fractured teeth. (A) Enamel bevelling; (B) V-shaped internal enamel groove; (C) Internal dentin groove; (D) External chanfer; (E) Overcontour and (F) Simple reattachment.*

prior to bonding, the precise fit between the segments may be lost, thus making the correct positioning of the fragment more difficult. This may explain why Franco and others (1985) and Davis, Roth and Levi (1983) suggested reattachment of the fragment prior to placing an *external chamfer* in the fracture line by means of a diamond round bur (Figure 1D), especially when the region corresponding to the fracture line is still evident after one week.

Chamfer placement has been performed on either the buccal (Reis & others, 2001) or lingual face (Silva Filho & Esberard, 1982). Others prefer a circumferential chamfer around the whole extension of the fracture line (Andreasen & others, 1991).

Overcontour

Although clinicians use the *overcontour technique* extensively, it is not commonly reported in the literature (Fahl & others, 1995; Fahl, 1997; Reis & others, 2001) (Figure 1E). After bonding the fragment, a superficial preparation (about 0.3-mm deep) is placed on the buccal surface using a cylindrical diamond-finishing bur extending about 2.5 mm coronally and apically from the fracture line. This area in then treated with a thin composite layer. This technique is useful when the fracture line is still evident after reattachment, overcoming this inconvenience with no extensive tooth preparation.

However, exposure of the resin composite to the oral environment using the chamfer and overcontour techniques may diminish the long-term esthetics due to the process of abrasion and discoloration that occurs over time with composites (Peumans & others, 1997; Andreassen & others, 1995). Placing a lingual chamfer minimizes this drawback, however, it may affect the wear rate of lingual surfaces.

Simple Reattachment

More recently, with improvements in hydrophilic adhesives, some authors have attempted to reattach fragments using *no additional preparation or simple reattachment* (Figure 1F) (Osborne & Lambert, 1985; Martens & others, 1988; Andreasen & others, 1993; Dickerson, 1994; Badami, Dunne & Scheer, 1995; Kanca, 1996; Pagliarini & others, 2000; Baratieri & others, 1998).

In addition to the assortment of techniques presented, some authors usually combine them. For example, the literature describes the association of a V-shaped internal groove and lingual enamel beveling (Simonsen, 1982) or placement of an internal dentin groove and the circumferential beveling of enamel margins (Baratieri & others, 1990; Burke, 1991).

What Reattachment Technique Should Be Chosen?

Several aspects may govern the choice of a reattachment technique. The research has reported that the primary cause of fragment loss is new dental trauma or the non-physiological use of restored teeth (Andreassen & others, 1995); therefore, most concerns about reattachment techniques have been directed toward their fracture strength. Therefore, it would be common to search for techniques that provide fracture strength similar to the one presented by sound teeth.

To date, few studies have attempted to evaluate the fracture strength of the reported techniques and their results vary considerably among researcher centers. Some authors have demonstrated reattachment techniques with fracture strengths of approximately 50% to 60% that of the intact tooth (Munksgaard & others, 1991; Worthington, Murchison & Vandewalle, 1999; Dean, Avery & Swartz, 1986).

De Santis and others (2001) compared simple reattachment with placement of a circumferential chamfer and concluded that the latter had higher fracture resistance when subjected to static and fatigue bending tests.

Reis and others (2001) have shown that simple reattachment recovered only 37.1% of intact tooth fracture resistance, while the buccal chamfer recovered 60.6%; and the overcontour and internal groove techniques nearly reached intact tooth fracture strength, recovering 97.2% and 90.5%, respectively.

The controversy among laboratory studies may result from methodological differences, for example, the mechanical test chosen, teeth origin, the method used to obtain tooth fragments and the fracture pattern and extension, among others. Regarding the method used to obtain the fragments, the authors have evidence (Loguercio & others, 2004) that different results were obtained when the teeth were fractured rather than cut with a thin diamond saw. When sectioning with a diamond saw, the reattachment techniques showed similar performance. In fact, most techniques presented a fracture strength recovery of approximately 60%. However, different fracture strengths were observed among the techniques when fractured instead of sectioned. Methods that promote tooth fracture are more realistic since they maintain precise fit between the remnant and the fragment that most uncomplicated crown fractures have. On the other hand, sectioning represents cases where the loss of structure was enough to avoid a precise fit between the parts. Therefore, this difference may be taken into account in the discussion of a paper, since the results may not be extrapolated for all fracture cases.

Unfortunately, the amount of strength recovery needed to keep the fragment in position long-term is not known. Perhaps fracture strengths as low as 50% to 60% may be sufficient. Further clinical studies must be conducted.

Other Factors Involved in the Choice of Techniques

Clinicians must also be aware of other aspects such as the need for endodontic therapy, fracture extension, fit

between fragment and the remaining tooth and fracture pattern before choosing the reattachment technique.

When endodontic therapy is required, the space provided by the pulp chamber may be used as an inner reinforcement, thus avoiding any excessive preparation of the fractured teeth (Amir, Bar-Gil & Sarnat, 1986; Diangelis & Jungbluth, 1992). In this case, esthetics is an important issue since pulpless teeth lose part of their translucency and brightness.

The *extension of the fracture* may also be a decisive choice. If the fracture extends close to the pulp horns and chamber, a direct pulp-capping agent that prevents placement of an internal groove in the remnant may be necessary. An option would be to place an internal groove only in the fragment (Baratieri & others, 1998). On the other hand, small enamel fragments should not be prepared before reattachment due to the friability of the enamel structure. In such cases, perhaps reattachment is impractical and only shaping the sharp edges and/or corrective grinding of the tooth would be sufficient. In other cases, reattachment may be done, however, only a simple reattachment would be possible (Baratieri & others, 1998).

The *quality of fit between segments* is an important factor to be considered. When the segments fit together with no discernible disruptions or defects, techniques that prevent resin composite from being exposed to the oral environment, such as placement of an internal groove, would be preferable except for simple reattachment, due to the low fracture strength recovery of this technique (Reis & others, 2001). Nevertheless, when enamel structure is lost in the trauma event, it may be more convenient to use an overcontour or chamfer technique (depending on the extension of the structure loss) so that the esthetics can be obtained simultaneously with the increase in adhesion area. There are some conditions where maintenance of the fragment in position can only be accomplished with special appliances developed exclusively to help clinicians use the fragments to restore the fractured teeth.

The *fracture pattern* is also an important factor to be considered. A review of published case reports indicates that 80% of traumatized incisors fracture in an oblique fashion from the labial to lingual aspects with the fracture line proceeding in an apical direction as seen in Figure 2B (Murchison & Worthington, 1998). This is an unfavorable fracture pattern that exhibits low resistance to labially applied forces (Dean, Avery & Swartz, 1986). This also occurs when fractures are approximately perpendicular to the long axis of the tooth (Figure 2C) (Dean & others, 1986). These fracture patterns have no lingual support and, therefore, are less resistant to labial forces.

According to Dean and others (1986), the most favorable fracture is when the plane of the fracture angles cervically in a lingual-to-labial direction (Figure 2A). This is explained by considering the amount of lingual support that the tooth provides the fragment when the fracturing force is placed on the facial aspect of the fragment.

Although the literature still lacks studies that evaluate reattachment techniques on specimens with different fracture patterns, it is advisable to choose techniques that provide high fracture strength recovery when dealing with unfavorable fracture patterns.

What Materials Should be Used for Fragment Reattachment?

The choice of materials varies among case reports. The development of more effective adhesive systems has encouraged clinicians to use only these materials to reattach fragments to the remnant (Munksgaard & others, 1991; Badami & others, 1995; Andreasen & others, 1993; Kanca, 1996; Liebenberg, 1997; Pagliarini & others, 2000).

Otherwise, other clinicians prefer to associate adhesive systems with other materials such as flowable composites (Small, 1996; Farik & Munksgaard, 1999; Farik & others, 1999; Farik, Munksgaard & Andreassen, 2000; Liebenberg, 1997), dual or chemically cured resin cements (Dickerson, 1994; Reis & others, 2001) and its light-cured version (Dean, Avery & Swartz, 1986). The use of viscous materials have been suggested where adhesive systems are used, along with hybrid and microfilled

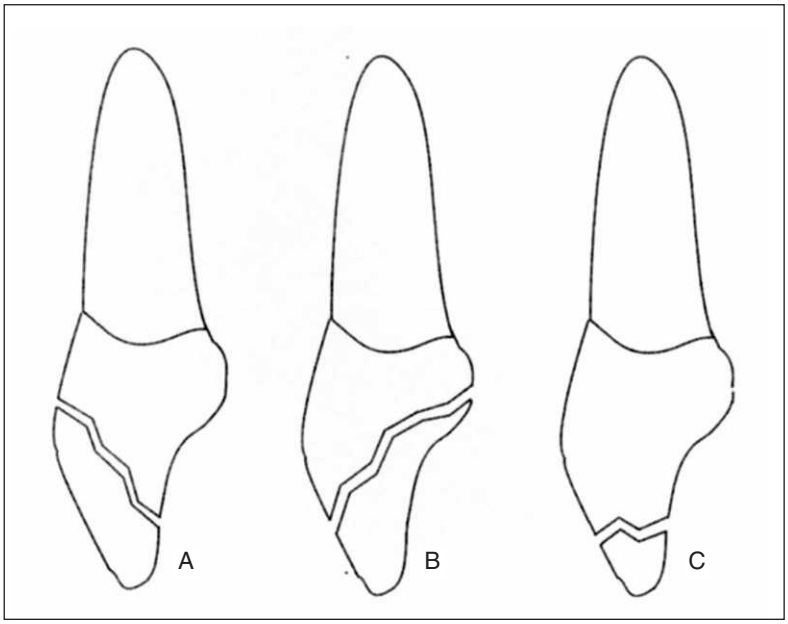


Figure 2. *Illustration of typical maxillary anterior fractured teeth. (A) Fracture line in an oblique fashion from the labial to lingual aspects in an apical direction. (B) Fracture line in an oblique fashion from the lingual to labial aspects in a cervical direction. (C) Fracture line approximately perpendicular to the long axis of the tooth. Adapted from Dean, Avery and Swartz (1986).*

light-cured resin composites (Martens & others, 1988; Simonsen, 1982; Burke, 1991; Diangelis & Jungbluth, 1992; Baratieri & others, 1994), as well as chemically cured resin composites (Dean & others, 1986).

As noted, many combinations of materials are reported in the literature and so far, only a few studies have evaluated their performance in terms of reattaching fragments to the remnant. As a result, several questions may arise: *Is only an adhesive system able to provide enough fragment retention? Does the association of materials improve the fracture strength of the reattachment? Is there any important interaction between the technique and the materials used for reattachment?* As mentioned above, only a few studies attempt to address these issues.

Most laboratory studies that have tested adhesive systems show that the kind of adhesive system used alters the fracture strength of the reattached teeth (Badami & others, 1995; Farik & others, 1998; Pagliarini & others, 2000) and is in the range of 40% to 60% of the fracture strength of sound teeth. Thus, the choice of adhesive system may depend on the clinician's experience with a specific material and the literature reports on its performance, such as bond strength values and the percentage of microleakage when tested *in vitro*.

A recent study has shown that the sole use of an adhesive system or its combination with higher mechanical property materials such as flowable resins, resin cements and resin composites have led to similar results when the fragment was reattached with no additional preparation (Reis & others, 2002). However, despite the statistical similarity, there was a trend toward improving fracture strength when the adhesive system was combined with a resin composite, which is likely due to the improved mechanical properties of this material compared to the others used.

The above study also tested the same materials; however, an additional preparation (buccal chamfer) was performed on the fracture line after reattachment and restored with a resin composite. Although no differences among the materials were detected, the fracture strength of these groups (chamfer technique) was superior to that obtained when no additional preparation was performed. These results suggest that the technique employed for reattachment is more important than the association of the materials.

Other reports have also reached similar conclusions. Oliveira (1994) reattached fragments using a chemically cured resin composite, a light cured resin composite and resin-modified glass ionomer cement with or without a bevel on the fracture line restored with a light cured resin composite. The authors concluded that the highest fracture strengths were obtained with the groups where a bevel was placed.

Similarly, Dean, Minutillo and Moore (1998) showed no difference in fracture strength when fractured teeth had their fragments reattached with only an adhesive system or when the adhesive system was associated with a light cured resin composite, a light-cured liner and a base of glass ionomer cement.

Some authors have attempted to use chemical cured resin composite or luting cements (Dickerson, 1994; Dean & others, 1986) in order to overcome the attenuation of light intensity that occurs when light activation is performed through the teeth (Loesche, 1999). In fact, using high light intensity along with higher exposure time can avoid inadequate polymerization. The combination of these two factors (light intensity and/or exposure time) may compensate for the attenuation of light intensity that occurs when light is applied over the tooth structure (Rueggeberg, Caughman & Curtis, 1994). In agreement with this finding, Dean and others (1986) did not detect differences between a light or chemical cured resin composite. Similarly, Reis and others, (2002) detected no difference when dual or light-cured resin cement was used.

The effect of material color change over time should be considered when chemical or dual versions of resin materials are used. For instance, consider enhancing esthetics. The amine accelerator necessary for dual polymerization can cause the color of resin cements to change over time (Rosenstiel, Land & Crispin, 1998). Since light-cured resin cements or composites are thought to be more color stable, they are preferable in cases where no additional preparation is to be done on the fracture line. The thin layer of chemical or dual cured resin material on the fracture line, exposed in the oral environment, may accelerate the color change.

Furthermore, recent studies have shown a certain degree of incompatibility between light-cured single-bottle adhesives and chemical-cured composites. The acidity that results from the presence of acidic resin monomers in light-cured single-bottle adhesive systems is likely responsible for the lower bond strength of chemical-cured composites applied over single-bottle adhesive systems (Sanares & others, 2001). These acidic resin monomers can react with tertiary amines (Lewis acid-base reaction). In turn, this affinity competes with the redox reaction that occurs with binary peroxide and amine catalytic components present in chemical-cured composites. Thus, the reduction of free radicals in single-bottle/chemical-cured resin interface will result in slow or no polymerization, depending on the acidity and concentration of acidic resin monomers (Sanares & others, 2001). A recent study that used dual-cured resin cement on simplified-step system further showed that the shear bond strength of the tested assembly was inversely related to the time interval between placement of the dual-cured resin cement and light activation (Schiltz & others, 2000). Competition

for the tertiary amine in red ox or acid-base reactions is in favor of the generation of free radicals; however, the prolonged contact with uncured acid resin monomers may interfere even with dual-cured composite materials.

Another disadvantage is that the manipulation of such materials requires dispensing equal amounts of base and catalyst pastes and proper mixing. This extra step in the manipulation of dual or chemical cured resin materials is also an inconvenience since it affects the degree of monomer conversion. Moreover, incorporating air-voids during mixing contributes significantly to reducing the mechanical properties of the polymerized material due to their inherent potential as stress-raisers within the chemical-cured resin cements or composites.

CONCLUSIONS

Several aspects govern the choice of a technique or the association of materials for fragment reattachment. However, the literature indicates that if the material or a combination of materials chosen has proven to be effective in *in vitro* studies and there is no incompatibility between them, the kind of material used for the reattachment of fractured teeth is less important. The authors do not advise clinicians to perform the simple reattachment of fractured teeth without additional preparation since this technique may not be able to restore even half of the fracture strength of intact teeth. Thus, clinicians should attempt to choose one of the reinforcement techniques related in this review in order to improve the fracture strength of the reattachment technique.

(Received 6 March 2003)

References

Amir E, Bar-Gil B & Sarnat H (1986) Restoration of fractured immature maxillary central incisors using the crown fragments *Pediatric Dentistry* **8(4)** 285-288.

Andreasen FM, Daugaard-Jensen J & Munksgaard EC (1991) Reinforcement of bonded crown fractured incisors with porcelain veneers *Endodontics & Dental Traumatology* **7(2)** 78-83.

Andreasen FM, Noren JG, Andreasen JO, Engelhardtsen S & Lindh-Stromberg U (1995) Long-term survival of fragment bonding in the treatment of fractured crowns: A multicenter clinical study *Quintessence International* **26(10)** 669-681.

Andreasen FM, Steinhardt U, Bille M & Munksgaard EC (1993) Bonding of enamel-dentin crown fragments after crown fracture. An experimental study using bonding agents *Endodontics & Dental Traumatology* **9(3)** 111-114.

Andreasen JO & Andreasen FM (1993) *Textbook and Color Atlas of Traumatic Injuries to the Teeth* Copenhagen Munksgaard 216-256.

Badami AA, Dunne SM & Scheer B (1995) An *in vitro* investigation into the shear bond strengths of two dentine-bonding agents used in the re-attachment of incisal edge fragments *Endodontics & Dental Traumatology* **11(3)** 129-135.

Bagheri J & Denehy GE (1983) Effect of enamel bevel and restoration lengths on Class IV acid-etch retained composite resin restoration *Journal of the American Dental Association* **107(6)** 951-952.

Baratieri LN, Monteiro S Jr & Andrada MAC (1998) *Esthetics: Direct Adhesive Restorations on Fractured Anterior Teeth* Chicago Quintessence Books, 135-205.

Baratieri LN, Monteiro S Jr & Andrada MAC (1990) Tooth fracture re-attachment: Case reports *Quintessence International* **21(4)** 261-270.

Baratieri LN, Monteiro S Jr, de Albuquerque FM, Vieira LC, de Andrada MA & de Melo Filho LC (1994) Reattachment of a tooth fragment with a "new" adhesive system: A case report *Quintessence International* **25(2)** 91-96.

Burke FJ (1991) Re-attachment of a fractured central incisor tooth fragment *British Dental Journal* **170(6)** 223-225.

Çaliskan MK & Türkün M (1995) Clinical investigation of traumatic injuries of permanent incisors in Izmir,Türkiye *Endodontics & Dental Traumatology* **11(5)** 210-213.

Cavallieri G & Zerman N (1995) Traumatic crown fractures in permanent incisors with immature roots: A follow-up study *Endodontics & Dental Traumatology* **11(6)** 294-296.

Chosack A & Eidelman E (1964) Rehabilitation of a fractured incisor using the patient's natural crown. Case report *Journal of Dentistry for Children* **31** 19-21.

Davis MJ, Roth J & Levi M (1983) Marginal integrity of adhesive fracture restorations: Chamfer versus bevel *Quintessence International* **14(11)** 1135-1146.

De Santis R, Prisco D, Nazhat SN, Riccitiello F, Ambrosio L, Rengo S & Nicolas L (2001) Mechanical strength of tooth fragment reattachment *Journal of Biomedical Materials Research* **55(4)** 629-636.

Dean JA, Avery DR & Swartz ML (1986) Attachment of anterior tooth fragments *Pediatric Dentistry* **8(3)** 139-143.

Dean JA, Minutillo AL & Moore BK (1998) A comparison of a hybrid light-cured glass ionomer base and liner vs a light-cured resin tooth fragment attachment *Pediatric Dentistry* **20(1)** 49-52.

Diangelis AJ & Jungbluth M (1992) Reattaching fractured tooth segments: An esthetic alternative *Journal of the American Dental Association* **123(8)** 58-63.

Dickerson WG (1994) Conservative re-attachment of a pulpally exposed fractured incisors *Dental Economics* **84(4)** 90-91.

Dietschi D, Jacoby T, Dietschi JM & Schatz JP (2000) Treatment of traumatic injuries in the front teeth: Restorative aspects in crown fractures *Practical Periodontal Aesthetic Dentistry* **12(8)** 751-758.

Ellis RG & Davey KW (1970) *Classification and Treatment of Injuries to the Teeth of Children* Chicago Year Book Medical 13-16.

Esberard RM, Silva Filho FPM & Gabrielli F (1978) [Caso clínico: Fratura coronária em dente anterior] *Revista da Associação Paulista dos Cirurgiões Dentistas* **32(2)** 130-134.

Fahl N Jr, Denehy GE & Jackson RD (1995) Protocol for predictable restoration of anterior teeth with composite resins *Practical Periodontics & Aesthetic Dentistry* **7(8)** 13-21 quiz 22.

Fahl N Jr (1997) Optimizing the esthetics of Class IV restorations with composite resins *Journal of the Canadian Dental Association* **63(2)** 108-111, 114-115.

Farik B, Munksgaard EC, Andreasen JO & Kreiborg S (1999) Drying and rewetting anterior crown fragments prior to bonding *Endodontics & Dental Traumatology* **15(3)** 113-116.

Farik B, Munksgaard EC & Andreasen JO (2000) Impact strength of teeth restored by fragment-bonding *Endodontics & Dental Traumatology* **16(4)** 151-153.

Farik B, Munksgaard EC, Kreiborg S & Andreassen JO (1998) Adhesive bonding of fragmented anterior teeth *Endodontics & Dental Traumatology* **14(3)** 119-123.

Farik B & Munksgaard EC (1999) Fracture strength of intact and fragment-bonded teeth at various velocities of the applied force *European Journal of Oral Sciences* **107(1)** 70-73.

Franco EB, Coradazzi JL, Ishikiriama A, Silva MH, Souza JR & Navarro MFL (1985) Re-attachment of a fractured fragment to a tooth, using the acid etch technique. A case report *Estomatologia e Cultura* **15** 47-50.

Hamilton FA, Hill FJ & Holloway PJ (1997) An investigation of dento-alveolar trauma and its treatment in an adolescent population. Part 1: The prevalence and incidence of injuries and the extent and adequacy of treatment received *British Dental Journal* **182(3)** 91-95.

Kanca J 3rd (1996) Replacement of a fractured incisor fragment over pulpal exposure: A long-term case report *Quintessence International* **27(12)** 829-832.

Liebenberg WH (1997) Reattachment of coronal fragments: Operative considerations for the repair of anterior teeth *Practical Periodontal & Aesthetic Dentistry* **9(7)** 761-772.

Loesche GM (1999) Marginal adaptation of Class II composite fillings: Guided polymerization vs reduced light intensity *Journal of Adhesive Dentistry* **1(1)** 31-39.

Loguercio AD, Mengarda J, Amaral R, Kraul A & Reis A (2004) Effect of fractured or sectioned fragments on the fracture strength of different reattachment techniques *Operative Dentistry* (in press).

Martens LC, Beyls HM, deCraene LG & D'Hauwers RF (1988) Reattachment of the original fragment after vertical crown fracture of a permanent central incisor *Journal of Pedodontics* **13(1)** 53-62.

Munskgaard EC, Hojtved L, Jorgensen EH, Andreassen JO & Andreassen FM (1991) Enamel-dentin crown fractures bonded with various bonding agents *Endodontics & Dental Traumatology* **7(2)** 73-77.

Murchison DF, Burke FJ & Worthington RB (1999) Incisal edge re-attachment: Indications for use and clinical technique *British Dental Journal* **186(12)** 614-619.

Murchison DF & Worthington RB (1998) Incisal edge reattachment: Literature review and treatment perspective *Compendium of Continuing Education* **19(7)** 731-734; 736; 738; 744.

Oliveira MDM (1994) [Estudo comparative *in vitro* de collagens de fragmento em incisivos superiores permanentes, através de quatro técnicas distintas] Master of Science Thesis Pediatric Dentistry Florianópolis Brazil.

Osborne JW & Lambert RL (1985) Re-attachment of fractured incisal tooth segment *General Dentistry* **33(6)** 516-517.

Pagliarini A, Rubini R, Rea M & Campese M (2000) Crown fractures: Effectiveness of current enamel-dentin adhesives in reattachment of fractured fragments *Quintessence International* **31(2)** 133-136.

Peumans M, Van Meerbeek B, Lambrechts P & Vanherle G (1997) The 5-year clinical performance of direct composite additions to correct tooth form and position I Esthetic qualities *Clinical Oral Investigation* **1(1)** 12-18.

Reis A, Francci C, Loguercio AD, Carrilho MR & Rodrigues Filho LE (2001) Re-attachment of anterior fractured teeth: Fracture strength using different techniques *Operative Dentistry* **26(3)** 287-294.

Reis A, Kraul A, Francci C, Assis TRG, Crivelli DD, Oda M & Loguercio AD (2002) Re-attachment of anterior fractured teeth: Fracture strength using different materials *Operative Dentistry* **27(6)** 621-627.

Rosenstiel SF, Land MF & Crispin BJ (1998) Dental luting agents: A review of the current literature *Journal of Prosthetic Dentistry* **80(3)** 280-301.

Rueggeberg FA, Caughman WF & Curtis JW Jr (1994) Effect of light intensity and exposure duration on cure of resin composite *Operative Dentistry* **19(1)** 26-32.

Sanares AM, Itthagarun A, King NM, Tay FR & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17(6)** 542-56.

Schiltz MY, Cincione F, Derijk W & Suh BI (2000) Bond strength of single bottle adhesives to self-cured composites on dentin *Journal of Dental Research* **79(Special Issue)** Abstract #1845 p 374.

Silva Filho FPM & Esberard RM (1982) [Restauração de dentes anteriores fraturados com aproveitamento de fragmentos] *Revista Gaúcha de Odontologia* **30** 99-103.

Simonsen RJ (1979) Traumatic fracture restorations: An alternative use of the acid etch technique *Quintessence International* **10(2)** 15-22.

Simonsen RJ (1982) Restoration of a fractured central incisor using original tooth fragment *Journal of the American Dental Association* **105(4)** 646-648.

Small BW (1996) Emergency reattachment of fractured tooth. Using dentin bonding agent and flowable composite *Oral Health* **86(10)** 33.

Spasser HF (1977) Repair and restoration of a fractured, pulpally involved anterior tooth: Report of case *Journal of the American Dental Association* **94(3)** 519-520.

Spinas E & Altana M (2002) A new classification for crown fractures of teeth *Journal Clinical Pediatric Dentistry* **26(3)** 225-232.

Starkey PE (1979) Reattachment of a fractured fragment to a tooth *Journal Indiana Dental Association* **58(5)** 37-38.

Tennery TN (1978) The fractured tooth reunited using the acid-etch bonding technique *Texas Dental Journal* **96(8)** 16-17.

Van der Vynver PJ & Marais JT (1996) Re-attachment of a fractured tooth fragment: A case report *Journal Dental Association of the South Africa* **51(10)** 623-624.

Walker M (1996) Fractured-tooth fragment re-attachment *General Dentistry* **44(5)** 434-436.

Worthington RB, Murchison DF & Vandewalle KS (1999) Incisal edge reattachment: The effect of preparation utilization and design *Quintessence International* **30(9)** 637-643.

Zerman N & Cavallieri G (1993) Traumatic injuries to permanent teeth *Endodontic Dental Traumatology* **9(2)** 61-64.

©Operative Dentistry, 2004, **29-2,** 234-237

Clinical Technique/Case Report

Resin Composite Reinforcement of Undermined Enamel

AA Abu-Hanna • IA Mjör

Clinical Relevance

The technique discussed enables the preservation of healthy but unsupported enamel. This technique may assist in optimizing the longevity of teeth.

SUMMARY

Studies have suggested that fracture resistance of undermined enamel increases when supported by a layer of bonded composite. Composite to reinforce enamel must have a secure foundation in dentin and/or enamel that is supported by dentin to perform optimally.

A restorative technique is presented using resin composite material to support and reinforce undermined enamel that lacks dentinal support in traditional amalgam restorations. This technique is intended to conserve unsupported enamel cavity walls and weakened cusps in extensive Class I and II preparations.

INTRODUCTION

The bonding properties of resin-based resin composite materials have allowed new concepts in cavity preparation design. *In vivo* and *in vitro* studies have been carried out to establish modern, conservative principles of cavity preparation. Furthermore, the current emphasis on the preservation of sound tooth structure calls for minimally invasive dentistry. Due to the evolution of resin composite materials and adhesive systems, the validity of several of GV Black's classical cavity preparation principles have been challenged. Principles such as "extension for prevention" and "the removal of unsupported enamel" are contradicted by evidence from current research in favor of preserving sound tooth structure.

Several studies have suggested that bonding the restoration to the remaining tooth structure increases the fracture resistance of permanent molars with MOD preparations (Denehy & Torney, 1976; Eakle, 1986; Watts, El Mowafy & Grant, 1987; Trope & Tronstad, 1991; Fissore, Nicholls & Yuodelis, 1991; Eidelman, 1999), thus reducing the risk of fracture, particularly of undermined enamel cusps. A study by Franchi, Breschi and Ruggeri (1999) showed that bonded amalgam appears to be as effective as bonded composite in supporting undermined enamel in terms of resistance to fracture. However, then concluded that composite may have better marginal adaptation to enamel compared to bonded amalgam and good marginal adaptation may be observed between amalgam and composite in composite–amalgam restorations. Composites may also be extended to replace more of the lost enamel to enhance the esthetic appearance of the restorations (Abu-Hanna & Mjör, 2004).

*Amer A Abu-Hanna, DDS, MS, assistant professor, Department of Operative Dentistry, College of Dentistry, University of Florida, Gainesville, FL, USA

Ivar A Mjör, BDS, MSD, MS, Dr odont, professor, Department of Operative Dentistry, College of Dentistry, University of Florida, Gainesville, FL, USA

*Reprint request: PO Box 100415, Gainesville, FL 32610, USA; e-mail: aabuhanna@dental.ufl.edu

Conserving unsupported enamel often may prevent subgingival tooth-restoration margins. Several studies confirmed the long held concept that restorations placed below the gingival margin (Schatzle & others, 2001), supragingival or gingival level margins (Yusof, 1991) are detrimental to gingival and periodontal health. Therefore, the use of contemporary resin composite materials and adhesive systems should be optimized with the aim of preserving all sound tooth structure.

METHODS AND MATERIALS

Amalgam restorations in stress bearing areas of posterior teeth have been universally accepted as the standard of care for many years. Their placement is not technique sensitive, they provide the least costly alternative of the directly placed restorations and they are durable. Amalgam has a wide range of indications and amalgam restorations serve well under a variety of conditions. However, they often require removing healthy tooth structure to accommodate their physical properties and removing unsupported enamel, which is considered weak and prone to fracture.

A variety of resin based, tooth colored direct restorative materials are available and they will be referred to as composites. These composites represent the only realistic alternative to amalgam for large, directly placed posterior restorations, but, for a long time after their introduction, they were not considered suitable for posterior restorations (Mjör & Wilson, 1998; Wilson & Mjör, 2000) mainly because of wear/degradation and polymerization shrinkage of the materials. Many teaching programs still hesitate to recommend large composite restorations in molars, despite improved physical properties of the materials.

Apart from color, some of the characteristics of present day composites include clinical properties similar to those of amalgam (Mjör, Moorhead & Dahl, 2000). However, contraction stresses still remain a major problem, even if incrementally inserted, especially for severely compromised teeth with weak, undermined cusps. When wide and deep cavity preparations with undermined enamel are restored with composites, they create considerable stress on the walls of the cavity preparation (Sakaguchi & others, 1991), and cuspal movements of up to 46 µm have been recorded (Suliman, Boyer & Lakes, 1994). Such stresses may lead to the fracture of undermined enamel, which can have a detrimental effect on the longevity of the restorations.

THE RESTORATIVE TECHNIQUE

The goal for this restorative technique is to combine the bonding and esthetic properties of resin based composite materials and the durability, wear resistance and strength of amalgam. This report illustrates two cases where composite is used to support undermined enamel, which normally would have to be removed. The remaining part of the cavity is then filled with amalgam.

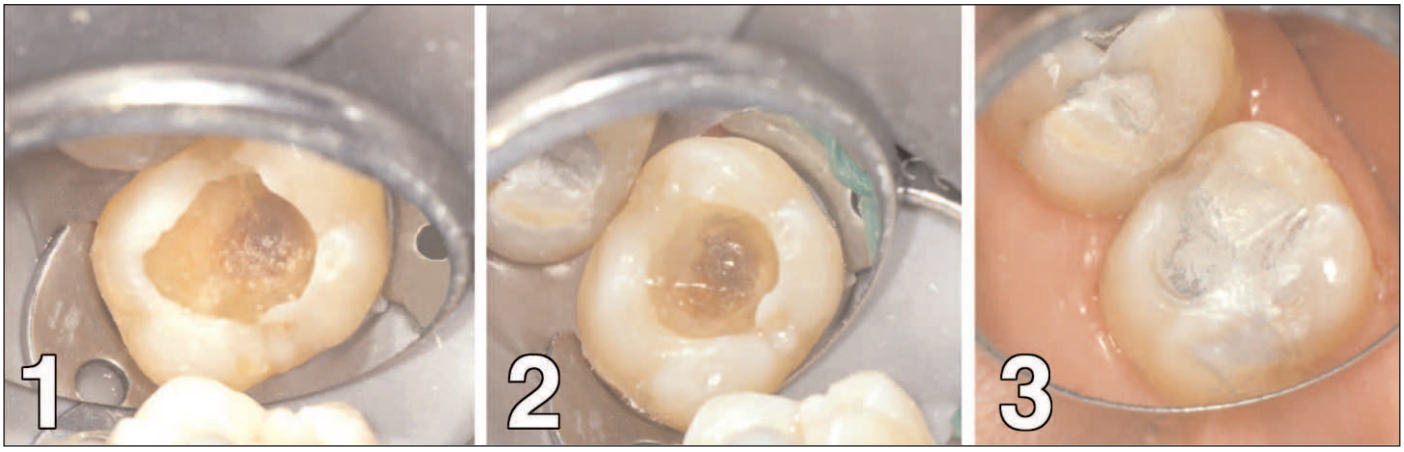
CASE REPORTS

Case 1

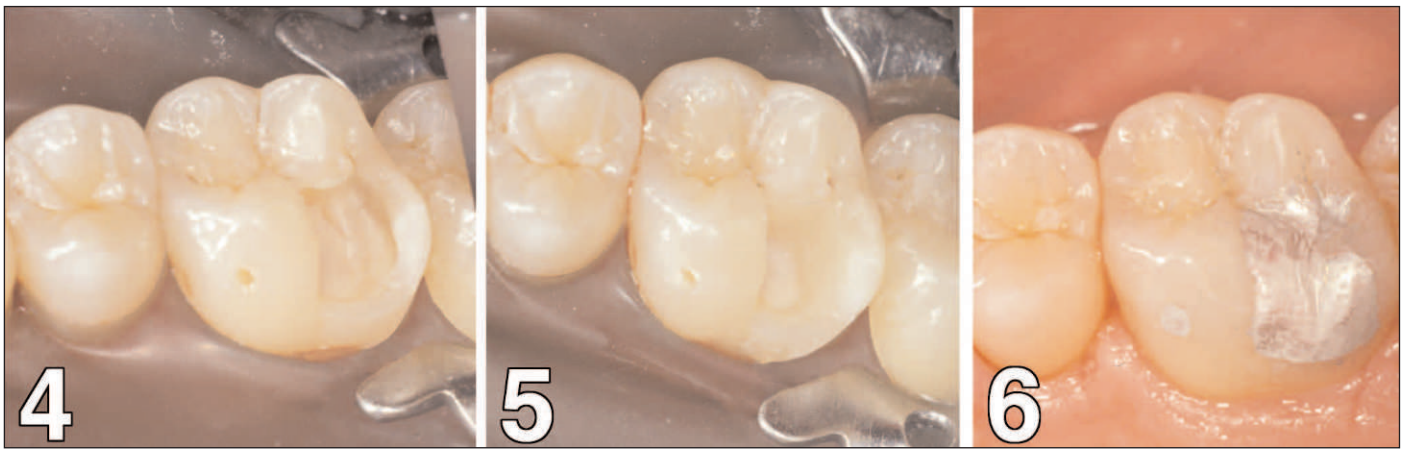
A second maxillary molar with gross occlusal caries was tentatively scheduled for an occlusal amalgam restoration. Radiographically, the depth and extent of the lesion was evident. The patient was informed that a final treatment plan for this tooth could be more accurately accomplished after caries removal and evaluation of the remaining tooth structure.

After cavity preparation and excavation of the carious lesion (Figure 1), the mesio-buccal and lingual cusps and mesial marginal wall comprised mainly enamel.

Traditionally, the preparation would extend to eliminate at least both lingual cusps and the mesial wall, followed by amalgam build up, including pins and a crown or endodontic treatment followed by a post, core and crown.



Case 1, Figures 1, 2 and 3.



Case 2, Figures 4, 5 and 6.

As an alternative treatment, the mesio-buccal and lingual cusps and mesial wall were bonded and lined with a hybrid composite (Feltic Z250, 3M/ESPE, St Paul, MN, USA) after conventional acid etching and bonding technique (Single Bond, 3M/ESPE) (Figure 2). The resin composite was added in one increment, 2-mm thick, one surface at a time and was applied from the cavosurface margin, extending 1 mm beyond the pulpo-lingual/mesial/buccal line angle to create a joint between the weak vertical walls of the preparation and the pulpal floor. A hybrid resin composite was the material of choice due to its superior physical properties compared to other composites.

The aim of this procedure was to replace the lost dentin and reinforce the remaining enamel. Subsequently, the occlusal preparation was restored with dispersed phase amalgam (Dispersalloy, Dentsply/Caulk, Milford, DE, USA) (Figure 3). In addition, occlusion was checked to verify the absence of any occlusal interferences.

Case 2

A large distal pit carious lesion in an upper first molar was scheduled for DOL amalgam restoration (Figure 4) after caries removal from the distal wall and the remaining part of the disto-lingual cusp consisted primarily of unsupported enamel. In addition, part of the disto-buccal cusp contained unsupported enamel. Due to the proximity of the pulpal floor to the pulp, Vitrobond glass ionomer liner cement (3M/ESPE) was placed to cover the deepest portion of the pulpal floor (Figure 4). Following the established principles for amalgam restorations, this preparation would have to eliminate the distal wall and unsupported enamel of the disto-buccal cusp, as it was considered weak and prone to fracture. As an alternative, the distal wall and disto-buccal cusp were bonded with composite using the same materials and techniques as in Case 1, leaving an occluso-lingual preparation with convergent walls (Figure 5). The cavity was then restored with a dispersed phase amalgam (Figure 6).

Over the last year, the authors have completed several restorations using this technique to reinforce unsupported enamel both in the student clinic and in private practice. Their experience with this type of restoration has been limited to this point and long-term data are not yet available. A six-month recall examination has indicated no problems. Furthermore, the patients have been instructed to report back if any part of the tooth fractures off and they will be recalled for routine check-up. To date, no problems have been reported.

If anyone adopts the illustrated restorative technique, the authors would appreciate learning of their experiences, preferably supported by clinical illustrations.

CONCLUSIONS

This technique uses resin composite material as dentin replacement to reinforce undermined enamel. After the enamel is reinforced, amalgam is used as the primary restorative material that provides strength, wear-resistance and the durability required in stress bearing areas. This technique will save tooth structure, minimize restoration size and surfaces and prevent the adverse effects of restoration margins and surfaces on periodontium.

(Received 22 April 2003)

References

Abu-Hanna A & Mjör I (2004) Combined amalgam and composite restorations *Operative Dentistry* **29(in press)**.

Denehy GD & Torney DL (1976) Internal enamel reinforcement through micromechanical bonding *Journal of Prosthetic Dentistry* **36(2)** 171–175.

Eakle WS (1986) Fracture resistance teeth restored with Class II bonded composite resin *Journal of Dental Research* **65(2)** 149–153.

Eidelman E (1999) Composite resin support of undermined enamel in amalgam restorations *Pediatric Dentistry* **21(2)** 118-120.

Fissore B, Nicholls J & Yuodelis R (1991) Load fatigue of teeth restored by a dentin bonding agent and a posterior composite resin *Journal of Prosthetic Dentistry* **65(1)** 80–85.

Franchi M, Breschi L & Ruggeri O (1999) Cusp fracture resistance in composite-amalgam combined restorations *Journal of Dentistry* **27** 47-52.

Mjör IA, Moorhead JE & Dahl JE (2000) Reasons for replacement of restorations in permanent teeth in general dental practice *International Dental Journal* **50** 361-366.

Mjör IA & Wilson NHF (1998) The teaching of Class I and Class II direct composite restorations in North America *Journal of the American Dental Association* **129** 1415-1421.

Sakaguchi RL, Sasik CT, Bunczak, MA & Douglas WH (1991) Strain gauge method for measuring polymerization contraction of composite restoratives *Journal of Dentistry* **19(5)** 312-316.

Schatzle M, Land NP, Anerud A, Boysen H, Burgin W & Loe H (2001) The influence of margins of restorations of the periodontal tissues over 26 years *Journal of Clinical Periodontology* **28(1)** 57-64.

Suliman AH, Boyer DB & Lakes RS (1994) Polymerization shrinkage of composite resins comparison with tooth deformation *Journal of Prosthetic Dentistry* **71(1)** 7-12.

Trope M & Tronstad L (1991) Resistance to fracture of endodontically treated premolars restored with glass ionomer cement or acid etch composite resin *Journal of Endodontics* **17(6)** 257–259.

Watts DC, El Mowafy OM & Grant AA (1987) Fracture resistance of lower molars with Class I composite and amalgam restoration *Dental Materials* **3(5)** 261–264.

Wilson NHF & Mjör IA (2000) The teaching of Class I and Class II direct composite restorations in European dental schools *Journal of Dentistry* **28** 15-21.

Yusof Z (1991) Proximal tooth surface quality and periodontal status *Journal of Oral Rehabilitation* **18(1)** 95-102.

Awards

American Academy of Gold Foil Operators
Distinguished Member Award

Dr J Martin Anderson

J Martin Anderson

It is a privilege and honor to be asked to stand before the members and guests of this esteemed group, the American Academy of Gold Foil Operators, and present the Distinguished Member Award to a dental school classmate. He deserves this recognition for all of the time, money and effort that he has given to the dental profession. He is far too humble to seek the limelight of success but quietly goes about his duties trying to make dentistry a better profession. It would not be a surprise to our fellow classmates that Jens Martin Anderson, affectionately known to his friends as Marty, would be the recipient of this award; rather, it would be a shock to them to know that I, of all people, would be the presenter. Wonders will never cease to amaze most people.

Our relationship goes back to the fall of 1961 when Marty and I began as freshmen at the University of Washington School of Dentistry. That has been so long ago that we can barely remember that we entered those hallowed halls with mixed emotions–worry, fear and anxiety. Many who started the class with us were gone by the time we graduated. I am sure that Marty thought I would be one of them. Marty was always a serious, dedicated student, and those traits followed him into private practice upon graduation from dental school in 1965. They have continued to serve him well throughout his life.

Marty began his long, illustrious teaching career at the University of Washington Dental School in 1968 as a clinical assistant. Now, he is on the part time faculty at the dental school while also maintaining a private practice. Marty has spent countless hours and some of his own money to develop excellent teaching manuals as well as serving as managing editor of *Operative Dentistry* from 1974 to 1999. He has also worked tirelessly to help raise more than $700,000 for various endowment funds for the dental school.

Marty has given numerous lectures, table clinics and chairside demonstrations before this group and others. He has been an active Gold Foil Study Club participant for more than 30 years. He has also reviewed published textbooks in addition to publishing several operative manuals and syllabi. To make his manuals more readable, Marty has included some cartoon characters for humor and some philosophical quotes for stimulation.

As a member of numerous professional and civic organizations, Marty has served as president of the Academy of Operative Dentistry, Associated Ferrier and the University of Washington Dental Alumni just to name a few. When his children were young, Marty was very active in Scouting and he and his family have always been very active in their church and its activities.

Marty has received many professional and dental school honors, which include membership or fellowship in OKU, the American Academy of Restorative Dentistry, the American and International College of Dentists, CAIC and many others. Furthermore, dental students and his peers have recognized him for his outstanding teaching and operative skills.

After all the above mentioned accomplishments, plus helping to raise three children with all of their activities, Marty still found time to pursue his passion—restoring and collecting antique cars. He has a shop for restoring these vehicles that would be the envy of many and is a member of a number of antique car clubs. The antique car aficionados have recognized Marty's meticulous work and attention to detail by awarding him first place honors in several meets such as the Pebble Beach Concours d'Elegance in 1985 and the Classic Car Club Grand Classic in 1997. Marty also finds time to create exquisite woodcarvings that stem from his appreciation for fine sculptures, crystal and art in addition to his love for classical music.

On the lighter side, Marty's stoic nature masks his dry Norwegian sense of humor. Some of his more memorable escapades include turning down Jay Leno when he wanted to buy one of his classic cars and almost blowing Jay Leno up while giving him a ride in one of his steam driven cars.

Marty also bought a Norse warrior statue, had it shipped from Chicago and uses it as a light standard for his circular driveway. It serves as the Norwegian signal that the house you are looking for, after a long night of imbibing at study club, is yours.

By now you have realized that Marty is a fine example of an outstanding dentist, teacher and an unselfish, giving, caring human being with a very subtle sense of humor. Like all great individuals, he is a person who leads by example.

It is my pleasure to award the 2002 Distinguish Members plague in 2003 to Dr J Martin Anderson. Marty, please accept this plaque with the Academy's congratulations and best wishes.

Warren K Johnson

Departments

Classifieds: Faculty Positions

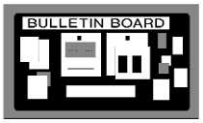
Operative Dentistry accepts appropriate classified advertisements from institutions and individuals. Advertisements are run at the following rate: $45.00 for 30 or fewer words, plus $0.75 for each additional word. Consecutively repeated ads are run at a flat rate of $50.00. ***Operative Dentistry*** 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.

The School of Dentistry
Oregon Health & Science University

The School of Dentistry at the Oregon Health & Science University is seeking an energetic, motivated and qualified individual for a full-time position at the level of assistant/associate professor in the Department of Restorative Dentistry, Division of Operative Dentistry. Experience in teaching, research, service and patient care, as well as excellent interpersonal and communication skills are preferred. Specific teaching responsibilities will include participation at the pre-doctoral level in both the clinical and pre-clinical curriculum, with the opportunity to serve as course director. Candidates should possess a DMD/DDS degree. One day per week (0.2 FTE) will be available for participation in the Faculty Dental Practice. Collaboration in research opportunities is available and encouraged. salary will be determined by credentials and experience. OHSU is an Equal Opportunity institution. Interested candidates should submit a letter, curriculum vitae and references to:

Dr Jack Ferracane, PhD
Chair, Restorative Dentistry
OSHU School of Dentistry
6111 SW Campus Drive
Portland, OR 97239-3097
(Ferracan@ohsu.edu)
(503) 494-4327

Announcements



Tucker Institute Course

A clinical course in conservative gold castings, mentored by Dr Richard V Tucker, will be held June 21-25, 2004 at the University of Washington Dental School in Seattle, Washington. During this five-day clinical course, you will prepare and seat at least four castings. You will be assisted in doing the lab procedures for one or two cases. Patients will be provided upon request. All lab fees, including gold costs, are covered in the course fee. Dental assistants will be provided. For course information, please contact Dr Dennis Miya, (206) 244-1618 or dmichi@aol.com.

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 of 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.

Authors: Complete instructions for contributors to the journal.

Reviewers: Link for our Editorial Board to submit manuscript reviews electronically.

Instructions to Contributors

Correspondence

Send manuscripts and correspondence regarding manuscripts to Dr Michael A Cochran, Editor, *Operative Dentistry*, Indiana University School of Dentistry, Room S411, 1121 W Michigan St, Indpls, IN 46202-5186; phone (317) 278-4800; fax (317) 278-4900; e-mail: editor@jopdent.org; URL: http://www.jopdent.org/.

Exclusive Publication

All material submitted for publication must be submitted exclusively to *Operative Dentistry*. Manuscripts not following the form outlined below may be returned for correction and resubmission.

Manuscripts

❒ Submit an original typed manuscript and three copies. The manuscript should include a short title for running headlines. Any identifying information (author's names, etc) should be on a separate page and not a part of the manuscript. Authors with English as a second language should consider having their manuscript reviewed for grammar, syntax and punctuation prior to submission.

❒ Submit a computer disk and identify the operating system (Macintosh or IBM-compatible) and the word processing program used.

❒ Identify the corresponding author and provide a complete address, fax number and e-mail address.

❒ Supply complete names, degrees, titles and affiliations for all authors (include addresses that are different from the corresponding author's).

❒ Proprietary names of equipment, instruments and materials should be followed in parenthesis by the name, company, city and state or country of the source or manufacturer.

❒ Research (clinical and laboratory) papers MUST include a one sentence Clinical Relevance statement, as well as a Summary, Introduction, Methods and Materials, Results, Discussion and Conclusions section. Funding other than material supply must be stated.

❒ Clinical Technique/Case Report papers should contain at least the following: Purpose, Description of Technique or Solution, along with materials and potential problems and a Summary outlining advantages and disadvantages.

❒ Type double-spaced, including references, and leave margins of at least 3 cm (1 inch). Spelling should conform to the *American Heritage Dictionary of the English Language*. SI (Système International) units are preferred for scientific measurement, but traditional units are acceptable.

❒ The editor reserves the right to make literary corrections.

Illustrations

Please do NOT submit any illustrations or graphs in Microsoft Power Point or Word format. They will not be accepted.

❒ Submit four copies of each illustration.

❒ Line drawings should be in India ink or its equivalent on heavy white paper, card or tracing velum. All lettering must be of professional quality, legible against its background and remain proportionally legible if reduced. Type legends on separate sheets.

❒ Photographs should be on glossy paper with a maximum size of 15x20 cm (6x8 inches). For best reproduction, a print should be one-third larger than its reproduced size.

❒ On the back of each illustration indicate lightly in pencil the top and the number of the figure ONLY (no names). Where relevant, state staining technique(s) and the magnification of the prints. Obtain written consent from holders of copyright to republish any illustrations published elsewhere.

❒ Illustrations may also be supplied on floppy disk, Zip disk or CD as TIFF files with a minimum resolution of 300 dpi (dots per inch) for grayscale and 1200 dpi for color.

❒ Photographs become the property of *Operative Dentistry*.

Tables and Graphs

❒ Submit tables and graphs on sheets separate from the text.

❒ Graphs are to be submitted with any lettering proportional to their size, with their horizontal and vertical axes values displayed.

❒ Data for constructing graphs MUST be provided with the manuscript in a spreadsheet (Excel) or word processing format on computer disk.

❒ Graphs may be supplied on floppy disk, Zip disk or CD as TIFF files with a minimum resolution of 300 dpi. or as Microsoft Excel files.

References

❒ References must be arranged in alphabetical order by authors' names at the end of the article, with the year of publication placed in parentheses immediately after the author's name. This is followed by the full journal title (no abbreviations and in italics), the full subject title, volume and issue number and first and last pages.

❒ In the text, cite references by giving the author and, in parentheses, the date: Smith (1975) found…; or, by placing both name and date in parentheses: It was found…(Smith & Brown, 1975; Jones, 1974).

❒ When an article being cited has three authors, include the names of all of the authors the first time the article is cited; subsequently, use the form (Brown & others, 1975). Four or more authors should always be cited in the text as (Jones & others, 1975). In the References section, always list all the authors.

❒ If reference is made to more than one article by the same author and is published in the same year, the articles should be identified by a letter (a, b) following the date, both in the text and in the list of references.

❒ Book titles should be followed by the publication address and the name of the publisher.

Reprints

Reprints of any article, report or letter can be ordered through the editorial office.

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.

DISCUS DENTAL®

SDI
SOUTHERN DENTAL INDUSTRIES

GC
GC America Inc.

DENTSPLY
CAULK

BISCO
Specialist In Adhesive and Composite Technology
Bringing Science to the Art of Dentistry™

ULTRADENT

3M ESPE

Den-Mat®

ivoclar vivadent

sds Kerr
SYBRON DENTAL SPECIALTIES

Please view the Corporate Sponsors Page at our website (http://www.jopdent.org) for direct links to these companies.

MARCH/APRIL 2004 • VOLUME 29 • NUMBER 2 • 121-240

EDITORIAL

CLINICAL RESEARCH

LABORATORY RESEARCH

LITERATURE REVIEW

CLINICAL TECHNIQUE/CASE REPORT

AWARDS

DEPARTMENTS

10-9385
Operative Dentistry
Indiana University School of Dentistry, Rm S411
1121 West Michigan Street
Indianapolis, IN 46202-5186 USA

Periodicals