The Quasi-Three-Dimensional Marginal Leakage of Full-Coverage Crowns: Resin Coating Versus Sodium Hypochlorite Treatment

Daizo Kawahara, DDS[a]/Atsushi Mine, DDS, PhD[b]/Jan De Munck, DDS, PhD[b]/Takuo Kuboki, DDS, PhD[c]/
Yasuhiro Yoshida, DDS, PhD[d]/Kazuomi Suzuki, DDS, PhD[e]/Bart Van Meerbeek, DDS, PhD[f]/
Hirofumi Yatani, DDS, PhD[g]

This study compared the effects of various surface treatments and techniques on the marginal leakage of full-coverage crowns using a quasi-three-dimensional evaluation. Crowns were cast using a gold-silver-palladium alloy by means of the lost-wax technique. Twenty-eight recently extracted human molars were divided randomly into four groups according to surface treatment before crown cementation: *(1)* no pretreatment (negative control), *(2)* primer (positive control), *(3)* resin coating and primer, and *(4)* phosphoric acid, sodium hypochlorite, and primer. All specimens were cemented with composite cement. The lowest marginal leakage was observed in group 4. Variation in marginal leakage between specimens originating from the same tooth was observed. *Int J Prosthodont 2010;23:406–409.*

In contrast to enamel, dentin is a much more challenging substrate to establish a reliable and durable bond. Different adhesive approaches, along with the specific adhesive composition and application procedure, influence the outcome considerably.[1] Despite the fact that adhesion to dentin has improved significantly, secondary caries caused by marginal leakage still remains a major problem.[2] Marginal leakage can not be controlled well, even when a high-precision casting method is employed and the restoration is luted adhesively. While some authors recommend the direct formation of a hybrid layer to achieve durable and strong bonding to dentin,[1] others have reported higher bond strengths when the surface was treated with sodium hypochlorite prior to cementation.[3] In this study, crown restorations were sectioned every 1 mm to check the marginal leakage in detail. The null hypothesis tested was that marginal leakage of full-cast crowns would not vary between the different dentin pretreatments.

Materials and Methods

Twenty-eight recently extracted human molars were prepared for full-cast crown restorations using a high-speed hand piece (NSK) with diamond points (K2 and K2ff, GC). Cervical margins were located 1 mm below the cementoenamel junction with a chamfer-type margin. All specimens were divided into four groups randomly according to their respective surface pretreatments: *(1)* no pretreatment (negative control), *(2)* primer (Panavia Fluoro Cement, Kuraray; positive control), *(3)* resin coating and primer (Clearfil SE, Kuraray), and *(4)* phosphoric acid, sodium hypochlorite, and primer (NaOCl; K-Etchant, Kuraray; AD Gel, Kuraray; Panavia Fluoro Cement) (Table 1). In the resin coating group, resin coating was applied on the abutment tooth and the margin was reprepared over an area approximately 1-mm wide using the K2ff bur to

---

[a] *Resident, Department of Oral and Maxillofacial Rehabilitation, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Science, Okayama, Japan.*

[b] *Postdoctoral Research Fellow, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Leuven, Belgium.*

[c] *Professor, Department of Oral and Maxillofacial Rehabilitation, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Science, Okayama, Japan.*

[d] *Associate Professor, Department of Biomaterials, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Science, Okayama, Japan.*

[e] *Professor, Department of Biomaterials, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Science, Okayama, Japan.*

[f] *Professor, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Leuven, Belgium.*

[g] *Professor, Department of Fixed Prosthodontics, Osaka University Graduate School of Dentistry, Osaka, Japan.*

Correspondence to: *Atsushi Mine, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Kapucijnenvoer 7, B-3000 Leuven, Belgium. Fax: +32-16-33.27.52. Email: amine@md.okayama-u.ac.jp*

Table 1 Materials Used per Experimental Group

Material	Composition	Application
Negative control group (no primer)	–	–
Positive control group with primer		
Panavia Fluoro Cement (Kuraray)	ED primer II A: HEMA, 10-MDP, 5-NMSA, water, accelerator; ED primer II B: 5-NMSA, water, sodium benzene; Paste A (universal): 10-MDP, 5-NMSA, silica, dimethacrylate monomer, photo-initiator, accelerator; Paste B (catalyst): barium glass, sodium fluoride, dimethacrylate monomer, BPO.	1. Mix ED primer II A and B. 2. Apply primer for 30 s and air dry. 3. Mix universal and catalyst paste for 20 s. 4. Light cure for 20 s. 5. Apply Oxyguard for 3 min.
Resin coating group with primer		
Clearfil SE (Kuraray)	Primer: 10-MDP, HEMA, hydrophilic dimethacrylate, photo-initiator, water; Bond: 10-MDP, HEMA, bis-GMA, hydrophilic dimethacrylate, photo-initiator, silanated colloidal silica.	1. Apply primer for 20 s and air dry. 2. Apply bond and gently air dry. 3. Light cure from occlusal side for 10 s. 4. Margin was reprepared over an area of almost 1 mm in width using a K2ff bur to get a fresh adhesive surface. 5. Impression of the abutment tooth.
Panavia Fluoro Cement	Same as for positive control group.	Same as for positive control group.
NaOCl treatment group with primer		
K-Etchant (Kuraray)	40% phosphoric acid, thickener	1. Apply for 30 s. 2. Rinse for 30 s.
AD Gel (Kuraray)	10% sodium hypochlorite, 14% aluminum oxide (alumina)	1. Apply for 60 s. 2. Rinse for 60 s.
Panavia Fluoro Cement	Same as for positive control group.	Same as for positive control group.

HEMA = hydroxyethyl methacrylate; 10-MDP = 10-methacryloyoxydecyl dihydrogen phosphate;
5-NMSA = N-methacryloyl-5-aminosalicylic acid; BPO = benzoyl peroxide; bis-GMA = bisphenol glycidyl methacrylate.

obtain a fresh adhesive surface before taking an impression. Impressions of the abutment teeth were then taken from specimens in all groups with an impression material (Exafine Injection Type, GC) and poured in die stone (Fujirock, GC) to produce a master cast. The dies were trimmed and covered with stone hardener (Aron Alpha, Toagosei), followed by two layers of die spacer (Ishifuku Material) above the preparation margin. The wax pattern (Inlay Wax medium, GC) was cast in a gold-silver-palladium alloy (Castwell M.C., GC). The fit of the castings was checked with silicone material (Fit Checker, GC) and the inner surfaces were air-abraded with 50-μm aluminum oxide.

After surface pretreatment, a dual-curing resin cement (Panavia Fluoro Cement) was mixed following the manufacturer's instructions and applied to the inside surface of the crown. The crown was seated in its terminal position using finger pressure and the excess resin cement was removed carefully. Oxiguard II (Kuraray) was applied along the margin area for 3 minutes for complete polymerization of the resin cement. Specimens were stored in 37°C distilled water for 24 hours.

All specimens were subjected to 2,500 thermal cycles (5°C and 55°C, 60 seconds each). The root surface was coated with two layers of nail varnish (NA, Shiseido) and specimens were stored in 0.2% fuchsine aqueous solution at 37°C for 24 hours. Subsequently, specimens were embedded in epoxy resin (Epofix, Struers) and sectioned using a low-speed diamond disk (Isomet, Buehler) into eight to nine slices (Fig 1). The length of marginal leakage was assessed for each slice using an optical microscope at a magnification of ×40 (CH30, Olympus).

Fabrication of full-cast crowns, surface treatment, restoration placement, and marginal leakage quantification were completed by one experienced operator. Data on marginal leakage were analyzed by one-way analysis of variance and the Scheffé test.

Results

The means and standard deviations of marginal leakage were 3.23 ± 0.11 mm for the negative control group, 2.06 ± 0.12 mm for the positive control group, 1.68 ± 0.14 mm for the resin coating group, and 0.68 ± 0.07 mm for the NaOCl treatment group (Fig 2). Significant differences in marginal leakage were recorded between all surface pretreatments, except between the positive control group and the resin coating group. Intraspecimen differences in marginal leakage were also observed (Fig 3).

Fig 1 Example of a series of cross-sections through a crown-restored tooth subjected to the marginal leakage test. **(left)** A typical section of a specimen showing marginal leakage up to 3-mm deep along the dentin-cement interface. **(right)** Eight to nine slices could be obtained from a single tooth.

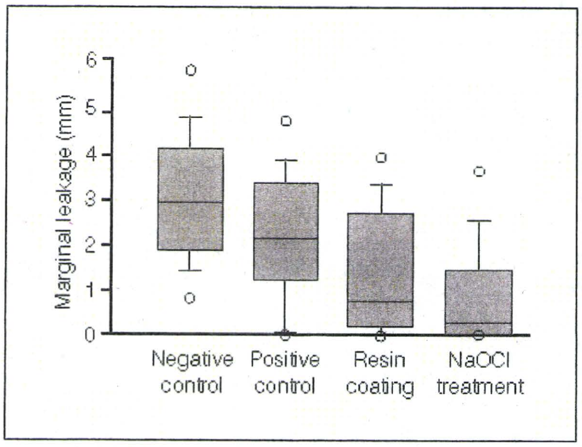

Fig 2 Box plot of mean marginal leakage per experimental group. Means connected with a horizontal line are not significantly different (*P* > .05, Scheffé test).

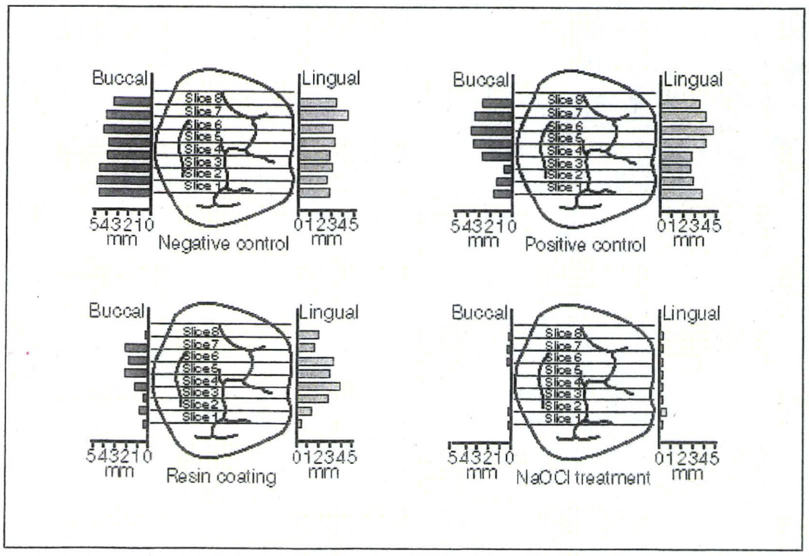

Fig 3 Marginal leakage measured at different sections through a crown-restored tooth for one tooth in each of the four different experimental groups. The horizontal bars on the left show the marginal leakage degree at the buccal aspect, and those on the right show the marginal leakage degree at the lingual aspect. In all specimens of the negative control group, leakage was detected (highest amount among all groups). No leakage was observed in some specimens of the positive control, resin coating, and NaOCl treatment groups.

Discussion

The null hypothesis that marginal leakage of full-cast crowns is not different for the four dentin surface pretreatments was rejected. In this study, marginal leakage decreased significantly, in order, in the negative control group, the positive control group, the resin coating group, and the NaOCl treatment group. Thus, the smallest marginal leakage was obtained when the surface was pretreated with sodium hypochlorite. Several studies have reported on the beneficial effect of removing the collagen layer by sodium hypochlorite prior to application of the adhesive resin.[3,4] Because almost all collagen fibrils are removed by sodium hypochlorite, no distinct hybrid layer is formed between the dentin and luting resin. Since resin infiltration is then expected to be poorer and lead to marginal leakage,[5] ED primer II (Kuraray) was used to produce a submicron hybrid layer that is typical of "mild" self-etching adhesives (pH: 2.1). This methodology has been shown to improve the marginal seal of the restoration.[4] Also, the technique's sensitivity is reduced since the critical step of drying the etched dentin surface with risks of over- or underdrying is avoided, as most of the collagen was removed from the surface beforehand. No significant difference in marginal leakage was found between the positive control group and the resin coating group. Since the margin was reprepared after resin coating over an area almost 1-mm wide but before taking the impression, this technique may have resulted in a similar surface effect as that obtained in the positive control group.

In the present study, finger pressure was used to seat the crown on the prepared tooth during cementation. Since the adherent surface was not flat but three-dimensional, the pressure for setting could be added properly to each specimen only through finger pressure. It was confirmed by observing the sections that the crowns were seated properly without tilting. Therefore, it can be estimated that the use of finger pressure for cementation did not affect the results significantly.

Surprisingly, big differences in marginal leakage were seen in the different sections originating from the same tooth (Fig 3). Traditionally, marginal leakage is assessed by slicing a restored tooth and scoring the leakage on the exposed surface only. This two-dimensional evaluation method, despite the leakage, is a three-dimensional phenomenon. Because of the variability of the results from this study, it is clear that marginal leakage analysis should be done at least in quasi-three dimensions, using several sections from the same tooth, if not in full three dimensions (eg, micro-CT technology).[6]

Conclusion

The formation of a reverse hybrid layer using sodium hypochlorite, followed by the application of a mild self-etching primer, results in the least amount of marginal leakage. Also, variations in marginal leakage between specimens originating from the same tooth were observed.

References

1. Van Meerbeek B, De Munck J, Yoshida Y, et al. Buonocore memorial lecture. Adhesion to enamel and dentin: Current status and future challenges. Oper Dent 2003;28:215–235.
2. Kidd EA. Diagnosis of secondary caries. J Dent Educ 2001;65:997–1000.
3. Kaneshima T, Yatani H, Kasai T, Watanabe EK, Yamashita A. The influence of blood contamination on bond strengths between dentin and an adhesive resin cement. Oper Dent 2000;25:195–201.
4. Prati C, Chersoni S, Pashley DH. Effect of removal of surface collagen fibrils on resin-dentin bonding. Dent Mater 1999;15:323–331.
5. Ferrari M, Mason PN, Vichi A, Davidson CL. Role of hybridization on marginal leakage and bond strength. Am J Dent 2000;13:329–336.
6. Jaecques SVN, Cardoso MV, Coutinho E, De Munck J, Van Meerbeek B. Sensitivity of micro-CT for microleakage detection around dental composite [abstract 0289]. J Dent Res 2008;87 (special issue C).

Journal of Oral Rehabilitation

Journal of Oral Rehabilitation 2010 **37**; 653–657

The effect of clinical experience on dentine bonding effectiveness: students versus trained dentists

M. UEDA*, A. MINE†, J. DE MUNCK†, T. HAKOGI*, B. VAN MEERBEEK† & T. KUBOKI* **Department of Oral Rehabilitation and Regenerative Medicine, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Science, Okayama, Japan, and †Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Leuven, Belgium*

SUMMARY Clinical successful application of dentine adhesives depends not only on material-related but also on operator-related factors. The purpose of this study was to evaluate the dentine bonding effectiveness of a self-etch composite cement applied by operators with or without clinical experience under well-standardized, randomized and blind conditions. Forty-eight bovine dentine surfaces were randomly divided into two groups. The first group consisted of eight dental students with no clinical experience at all, and the second group consisted of eight dentists with extensive experience in adhesive dentistry (mean experience of 11·4 years). Next, a 4-mm-diameter stainless steel rod (SUS-304) was bonded to the dentine surface using Panavia Fluoro cement (Kuraray Medical Inc., Tokyo, Japan). After application procedures, the specimens were randomized and shear bond-strength measurements were performed by a single blinded operator. Mann–Whitney *U* test was used to determine statistical differences in bond strength between the two groups, and Kruskal–Wallis was used to determine statistical difference between the student and dentist groups. The means and standard deviations of bond strength were 11·5 ± 8·1 MPa for the student group and 7·1 ± 4·3 MPa for the dentist group, respectively. The bond strength of the student group was significantly higher than that of the dentist group. However, the variability in bond strength was significantly higher in the student group, and some specimens failed prior to actual testing (included as 0 MPa). Clinical experience did not have a positive effect on the bonding effectiveness of the self-etch composite cement to dentine.

KEYWORDS: technique sensibility, operator variability, adhesion, shear bond strength, dentine bonding, adhesive resins, self-etching primer, bias control

Accepted for publication 19 March 2010

Introduction

Adhesion to dentine is regarded as more challenging and technique sensitive, mainly because of the wet nature of the substrate. Bonding to enamel is on the contrary considered adequate since very long (1). The best way to determine the effectiveness of dental adhesives is by randomized, controlled clinical trials. Such studies, however, last very long, are expensive and very often do not allow distinguishing the actual contribution of the adhesive procedures from the final clinical outcome. On the other hand, *in vitro* bond-strength testing enables to screen adhesives fast on their bonding effectiveness to a dental substrate, eventually in the hope to give an idea of their potential clinical effectiveness. Furthermore, because bonding procedures involve multiple steps, all prone to different errors, the final outcome is not only dependent on material-related factors, but also dependent on the performance of the individual operator (2). Very few reports are today available regarding such technique-sensitivity issues, especially with regard to the level of experience of the clinicians. Fundingsland *et al.* noticed considerable operator variability during the develop-

doi: 10.1111/j.1365-2842.2010.02095.x

ment of an experimental bonding system and concluded that bond strength was significantly influenced by the technique variability among the different operators (3). Nevertheless, to estimate the influence of clinical experience on bonding effectiveness, a study should be designed in such a way to rule out potential masking factors as for example experience in making bond-strength specimens.

The purpose of this study was to evaluate the effect of clinical experience on the bonding effectiveness of a self-etch composite cement to dentine. The null hypothesis tested was that clinical experience does not affect dentine bonding effectiveness.

Materials and methods

Tooth preparation

Forty-eight bovine mandibular incisors, frozen immediately after extraction, were used in this experiment. After defrosting, any soft tissue around the cervical area was removed, the radicular portion was cut and the pulp tissue removed. The vestibular side of the coronal portion was then ground flat using a model trimmer* to expose dentine. The teeth were positioned in a silicon mould, embedded in slow-curing epoxy resin† and polymerized for eight hours at room temperature. The exposed dentine area was polished using a 600-grit silicon carbide waterproof abrasive paper under running water using an automatic polishing machine‡.

All tooth preparations were performed by a single operator (MU); all specimens were randomly divided into two groups.

Dentine bonding procedure

Two groups, each consisting of eight examinees, were selected as follows:

The student group consisted of eight undergraduate third-year students from Okayama University Dental School. All of them did not have any experience, either in making bond-strength test specimens or in using dental adhesives. They actually never used adhesives, not even during the dental courses, and therefore should be considered as having no (clinical) experience at all. The dentist group consisted of eight dentists, practicing at Okayama University Hospital Clinical Division in Dentistry. All of them did not have any experience in making bond-strength test specimens. The range of clinical experience varied from 6·5 to 17·5 years (with a mean clinical experience of 11·4 years).

All examinees were instructed on how to make bond-strength specimens. Handling procedures for the adhesive cement were not explained, but the manufacturer's instructions (i.e. the primer has to be dried completely) with some extra pictures were provided (Fig. 1). The adhesive resin used was Panavia Fluoro cement.§ Panavia Fluoro Cement is similar to Panavia F§ except that the ED Primer is replaced by ED Primer II (Table 1). ED primer II had to be applied on dentine with a microbrush and left untouched for 30 s and subsequently air-dried with oil-free compressed air from an air syringe. Next, a 4-mm-diameter stainless steel rod (SUS-304), sandblasted using 50-μm alumina, was cemented onto the dentine surface by finger pressure with Panavia Fluoro cement§. Oxyguard II was applied around the transition of dentine to the stainless steel rod for 3 min to block out oxygen. The bond-strength specimens were kept in water at 37 °C for 24 h.

Shear bond-strength measurement

Next, all specimens were randomized by a single operator (MU) and blinded for the operators who had prepared the specimens. The actual bond-strength measurements were performed by another operator (TH), as follows: the shear bond strength of the 48 specimens in each group was measured using a universal testing machine¶ at a crosshead speed of 1 mm per minute. When specimens failed before actual testing, a bond strength of 0 MPa was included in the calculation of the mean Shear bond-strength.

Statistical analysis

Mann–Whitney *U* test was used to determine statistical differences in bond strength between the two groups at a significance level of 0·05. Kruskal–Wallis was used to determine statistical differences in the student and dentist groups.

---

*MT-6; J. Morita Co., Tokyo, Japan.

†Epofix, Struers, Copenhagen, Denmark.

‡Automet 2/Ecomet 3; Buehler, Lake Bluff, IL, USA.

§Kuraray Medical Inc., Tokyo, Japan.

¶AUTOGRAPH/AGS-500D, Shimadzu, Japan.


接着試験手順

はじめに

セメント練和

歯面処理

2個目以降の試料は以下の操作を繰り返します

合着

除去前

除去後

2個目以降の試料は歯面処理から行ってくだ

Fig. 1. Instruction sheet. An instruction card was given to the examinees, explaining handling procedures for the self-etch cement and the method how to make bond-strength specimens.

Table 1. Material used

Material	Composition	Application
Panavia fluoro cement (Kuraray Medical Inc., Tokyo, Japan)	ED Primer II A: HEMA, 10-MDP, 5-NMSA, water, accelerator ED Primer II B: 5-NMSA, water, sodium benzene Paste A (universal): 10-MDP, 5-NMSA, silica, dimethacrylate monomer, photo-initiator, accelerator Paste B (catalyst): barium glass, sodium fluoride, dimethacrylate monomer, BPO	Mix ED primer II A and B Apply Primer for 30 s and air-dry Mix universal and catalyst paste for 20 s Apply Oxyguard for 3 min

HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; 5-NMSA, N-methacryloyl-5-amino-salicylic acid.

Results

The mean bond strength and standard deviation was 11·5 ± 8·1 MPa for the student group and 7·1 ± 4·3 MPa for the dentist group, respectively (Fig. 2). The bond strength of the student group was significantly higher than that of the dentist group ($P = 0{\cdot}0145$). Variability was on the other hand much higher in the student group. For example, in the student group some of the bond-strength specimens failed before actual testing. Using Kruskal–Wallis, statistical difference ($P = 0{\cdot}0208$) was recorded among the examinees in the student group (Fig. 3), while not in the dentist group ($P = 0{\cdot}1694$).

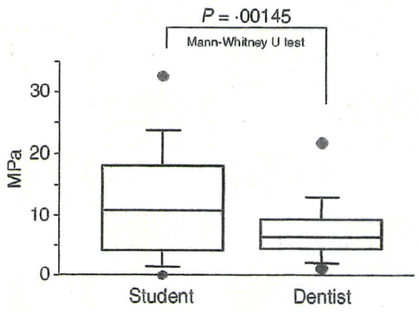

Fig. 2. Bond-strength data of the student and dentist groups. The box represents the spreading of the data between the first and the third quartile. The central line represents the median. The whiskers denote the range of variance, and the outliers represent the maximum and minimum. The mean bond strength of the student group was significantly higher than that of the dentist group.

Discussion

The null hypothesis that clinical experience does not affect dentine bonding effectiveness was rejected. These results suggest that clinical experience is not a prerequisite for achieving adequate bonding effectiveness. Otherwise, a known problem in assessing bond strength is that materials are never tested under standardized conditions. Not only is there a discrepancy between the actual bond-strength values for one and

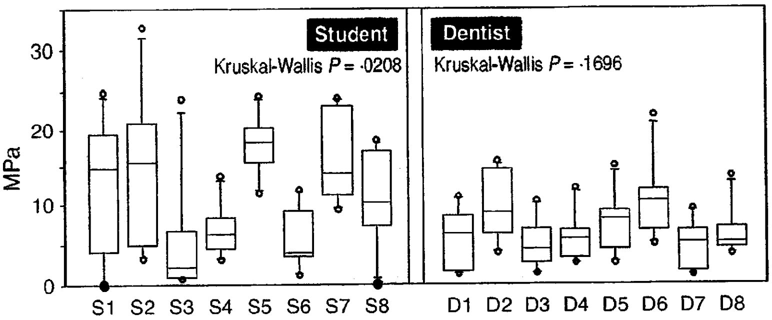

Fig. 3. Individual bond-strength data. Box plot of the data from each examinee, showing a large variety in bond strength between the operators. The student group revealed a statistical difference among the examinees. Only in the student group, some of the specimens failed before actual testing and were included as 0 MPa.

the same product when measured at different laboratories worldwide, but also the standard deviations vary widely. Such high variance in bond strengths may result from either differences in the nature of the sample or differences in the experimental protocol. In particular, the skills and the experience of the operator cannot be analysed and may be an important source leading to widely varying results (4), as was also shown in this study. The coefficient of variation [CV (%), (standard deviation/mean) × 100)] expresses the variation seen in data relative to size of the mean, and it is useful because it allows comparison of the variation in one data set to that in another (5). Scherrer *et al.* reported an average CV between 24% and 49%, obtained from 147 shear bond-strength test studies (6). In this study, the CV was 71% for the student group and 61% for the teacher group, respectively. We excluded the experience in the fabrication of bond-strength test specimens, as none of the examinees did have experience in performing bond-strength tests. This inexperience may explain some of the high variability. However, as mentioned earlier, also substrate differences can contribute to this high variability, but they were ruled out as much as possible in this study, first by having used a high number of teeth per group, secondly by having applied an adequate randomization protocol and thirdly by having blinded the operator who measured the bond strength to the examinee.

Very few reports are today available on the bonding effectiveness being dependent on the clinical experience of the clinician. Sano *et al.* reported that the tensile bond strength of the self-etch adhesive Clearfil Liner Bond II[§] showed no statistically significant difference between the student and dentist groups (7). However, in their study, the dentists were full-time member of the Department of Operative Dentistry of Tokyo Medical and Dental University, and all of them had extensive research experience in adhesive dentistry, which is probably more helpful than clinical experience alone. Miyazaki *et al.* also reported on the shear bond strengths of a self-etch adhesive** to dentine, when applied by three different operator groups (5th-year dental students, dental practitioners with a mean clinical experience of 18·1 years, but without any experience in bond-strength testing, and university dentists with a mean clinical experience of 5·1 years as well as experience in bond-strength testing) (6). In this study, no statistical difference between the students and dental practitioners was observed, but the university dentists scored significantly better than all others. Therefore, extensive knowledge of the science behind adhesives seems more important than clinical experience.

Surprisingly, the mean bond strength of the student group in our study was significantly higher than that of the dentist group. It takes 6 years to graduate from a Japanese dental school. In Okayama University Dental School, the latter half of their fifth and the earlier half of their sixth years are periods of clinical training. The examinees of the student group were third-year students and therefore had no experience with dental adhesives, not only clinically, but also theoretically. Miyazaki *et al.* reported that dental students using dental adhesives for the first time tended to read instructions more carefully and to make the bonding specimens more meticulously step by step (8). Therefore, the inexperience can apparently turn into an advantage, as also observed in this study. Giachetti *et al.* evaluated the influence of the operator skills on microleakage of Class-V restorations using four types of adhesives[††] (9, 10). The microleakage score for the

---

[§] Kuraray Medical Inc., Tokyo, Japan.

** Fluoro Bond, Shofu, Kyoto, Japan.

[††] Prime&Bond NT, Dentsply, Konstanz, Germany; AdheSE, Ivoclar-Vivadent, Schaan, Liechtenstein; Adper Scotchbond 1XT, 3M ESPE, Seefeld, Germany; Adper Prompt L-Pop, 3M ESPE.

one-step self-etch adhesive†† was better for the student group than for expert group (10).

The operator (TH), who was blinded to the examinees and measured the bond strength, did not detect any specimens that visually exhibited defects because of inaccurate preparation. Hence, all specimens appeared to have been prepared with the needed carefulness. Special care was taken that no residual cement remained between the tooth surface and the stainless steel rod. When observing the application procedures, the other operator (MU) discovered two typical differences in application procedures used by the two groups. At first, students appeared not good at mixing the cement, so that the cement must have been less mixed in this group, while the actual mixing time was the same in both groups. The second difference observed was that students used more the maximum power of the air syringe, while the dentists tended to more mildly air-dry the self-etch primer. The dentists involved in this study had used the Panavia luting cements in daily clinics for a long time. In the past, mild air-blowing was deemed important. For self-etch adhesives, air-drying /thinning of the primer is known to have a significant effect on the dentine bond strength (11–13). Under-drying may result in incomplete evaporation of the primer solvents and so under-polymerization of the bonding agent (8).

Despite the higher mean bond strength in the student group, some specimens failed before actual testing (=pre-testing failure). In a clinical situation, it is maybe more important to have consistent results rather than a high mean value, but with some specimens that did not bond at all. Therefore, it is not sure that the results obtained by the students are better than those obtained by the dentists. Further research is needed to clarify in more detail all factors that can contribute to these technique-sensitivity issues.

Conclusion

In dentistry, there has been a myth that clinical successful application of dentine adhesives depends on the operator experience. The dentine bonding effectiveness of the self-etch composite cement applied by operators with or without clinical experience was evaluated under well-standardized, randomized and blind conditions. Clinical experience seems not necessarily to be a prerequisite to achieve good bonding effectiveness to dentine using self-etch adhesives.

References

1. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P *et al.* Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. Oper Dent. 2003;28:215–235.
2. Jacobsen T, Söderholm KJ. Effect of primer solvent, primer agitation, and dentin dryness on shear bond strength to dentin. Am J Dent. 1998;11:225–228.
3. Fundingsland JW, Aasen SM, Ario PD, Glasspoolc EA. Investigations into technique variability in bonding studies. J Dent Res. 1996;75:126.
4. Leloup G, D'Hoore W, Bouter D, Degrange M, Vreven J. Meta-analytical review of factors involved in dentin adherence. J Dent Res. 2001;80:1605–1614.
5. Woolson RF. Statistical methods for analysis of biomedical data. New York (NY): John Wiley & Sons, 1987:23–24.
6. Scherrer SS, Cesar PF, Swain MV. Direct comparison of the bond strength results of the different test methods: a critical literature review. Dent Mater. 2010;26:e78–e93.
7. Sano H, Kanemura N, Burrow MF, Inai N, Yamada T, Tagami J. Effect of operator variability on dentin adhesion: students vs. dentists. Dent Mater J. 1998;17:51–58.
8. Miyazaki M, Onose H, Moore BK. Effect of operator variability on dentin bond strength of two-step bonding systems. Am J Dent. 2000;13:101–104.
9. Giachetti L, Scaminaci Russo D, Bertini F, Pierleoni F, Nieri M. Effect of operator skill in relation to microleakage of total-etch and self-etch bonding systems. J Dent. 2007;35:289–293.
10. Giachetti L, Scaminaci Russo D, Bambi C, Nieri M, Bertini F. Influence of operator skill on microleakege of total-etch and self-etch bonding systems. J Dent. 2008;36:49–53.
11. Miyazaki M, Platt JA, Onose H, Moore BK. Influence of dentin primer application methods on dentin bond strength. Oper Dent. 1996;21:167–172.
12. Finger WJ. Dentin bonding agents. Relevance of *in vitro* investigations. Am J Dent. 1988;1:184–188.
13. Spreafico D, Semeraro S, Mezzanzanica D, Re D, Gagliani M, Tanaka T *et al.* The effect of the air-blowing step on the technique sensitivity of four different adhesive systems. J Dent. 2006;34:237–244.

Correspondence: Atsushi Mine, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Kapucijnenvoer 7, B-3000 Leuven, Belgium.
E-mail: amine@md.okayama-u.ac.jp

Journal of Dental Research
http://jdr.sagepub.com/

Enamel-Smear Compromises Bonding by Mild Self-Etch Adhesives

A. Mine, J. De Munck, M. Vivan Cardoso, K.L. Van Landuyt, A. Poitevin, T. Kuboki, Y. Yoshida, K. Suzuki and B. Van Meerbeek

J DENT RES 2010 89: 1505 originally published online 7 October 2010
DOI: 10.1177/0022034510384871

RESEARCH REPORTS
Biomaterials & Bioengineering

A. Mine[1], J. De Munck[1], M. Vivan Cardoso[1], K.L. Van Landuyt[1], A. Poitevin[1], T. Kuboki[2], Y. Yoshida[3], K. Suzuki[3], and B. Van Meerbeek[1]*

[1]Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Kapucijnenvoer 7, B-3000 Leuven, Belgium; [2]Department of Oral Rehabilitation and Regenerative Medicine, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, Okayama, Japan; and [3]Department of Biomaterials, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Science, Okayama, Japan; *corresponding author, bart.vanmeerbeek@med.kuleuven.be

J Dent Res 89(12):1505-1509, 2010

Enamel-Smear Compromises Bonding by Mild Self-Etch Adhesives

ABSTRACT

In light of the increased popularity of less acidic, so-called 'ultra-mild' self-etch adhesives, adhesion to enamel is becoming more critical. It is hypothesized that this compromised enamel bonding should, to a certain extent, be attributed to interference of bur debris smeared across enamel during cavity preparation. High-resolution transmission electron microscopy revealed that the enamel smear layer differed not only in thickness, but also in crystal density and size, depending on the surface-preparation method used. Lab-demineralization of sections clearly disclosed that resin-infiltration of an ultra-mild self-etch adhesive progressed preferentially along micro-cracks that were abundantly present at and underneath the bur-cut enamel surface. The surface-preparation method significantly affected the nature of the smear layer and the interaction with the ultra-mild adhesive, being more uniform and dense for a lab-SiC-prepared surface *vs.* a clinically relevant bur-prepared surface.

KEY WORDS: ultra-mild self-etch adhesive, adhesion, resin-smear complex, enamel, TEM, smear layer, hybrid layer.

DOI: 10.1177/0022034510384871

Received March 4, 2010; Last revision May 16, 2010; Accepted May 24, 2010

A supplemental appendix to this article is published electronically only at http://jdr.sagepub.com/supplemental.

INTRODUCTION

In 1968, Buonocore *et al.* introduced the so-called 'acid-etch technique' to micro-mechanically interlock resin within enamel upon impregnation into the acid-produced etch pits (Buonocore, 1955; Buonocore *et al.*, 1968). Today, this technique is still the most effective for reliable and durable bonding to enamel (Van Meerbeek *et al.*, 2003). Recently, self-etch adhesives were introduced to fulfill clinicians' demands for simple, fast, and low-technique-sensitive bonding procedures. To overcome the shelf-life problems that are commonly associated with one-step adhesives (Salz *et al.*, 2005; Nishiyama *et al.*, 2006), so-called 'ultra-mild' self-etch adhesives are significantly less acidic (pH ≈ 2.7). They interact only very superficially with the tooth surface (Koshiro *et al.*, 2006; Sarr *et al.*, 2009). Clinically, composite restorations bonded with a mild or 'ultra-mild' self-etch adhesive typically suffer from marginal adaptation problems at the enamel surface (Peumans *et al.*, 2007).

It is hypothesized that the compromised enamel bonding with (ultra-)mild self-etch adhesives should, to a certain extent, be attributed to interference of bur debris smeared across enamel during cavity preparation. Somewhat surprisingly, very little information is available on the morphology of enamel smear-layers (Hannig *et al.*, 2002). Therefore, the purpose of this study was to examine the effects of different surface-preparation methods on the interfacial structure of an ultra-mild self-etch adhesive bonded to enamel by high-resolution transmission electron microscopy.

MATERIALS & METHODS

Non-carious human third molars were stored in 0.5% chloramine solution at 4°C and used within 1 mo after extraction. The teeth were randomly divided into 3 groups. The lingual/buccal enamel of a first set of teeth was flattened by means of a medium-grit (100 µm) diamond bur (842, Komet, Lemgo, Germany) in a water-cooled high-speed contra-angle handpiece mounted in the MicroSpecimen Former (The University of Iowa, Iowa City, IA, USA) (Bur-cut group). In half of these, the bur-cut enamel surface was further polished by wet-sanding with #600-grit silicone-carbide paper (SiC-ground group). From the third set of teeth, the lingual/buccal surface was cleaned with pumice by means of a soft-bristle brush mounted in a handpiece to

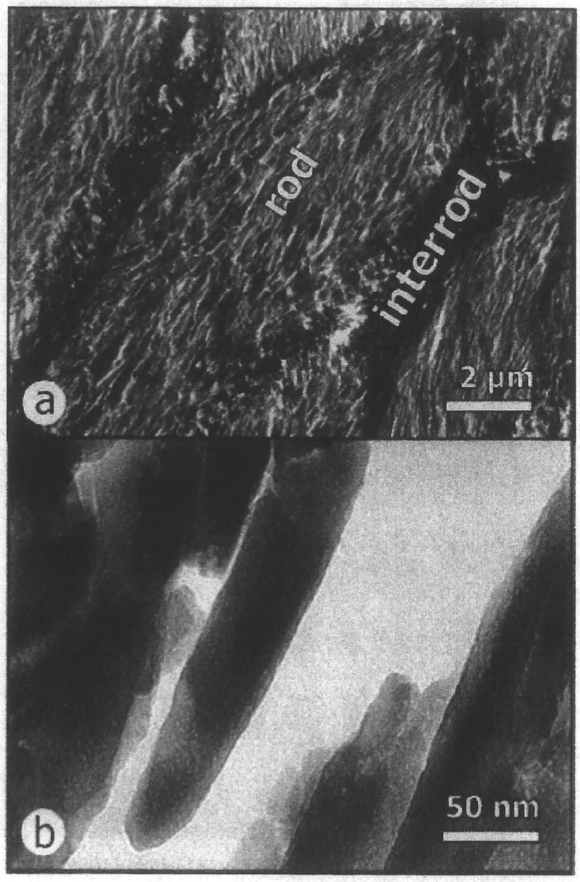

Figure 1. Micro- and nano-structure of enamel. **(a)** The electron density at the rod periphery (inter-rod) is denser than that of the center. The diameter of enamel rods is about 4 µm in this section. A tightly packed crystal structure and the variation of crystal orientations within the enamel rod can be observed. Inter-rod enamel appears denser, but less structured. **(b)** An ultra-high magnification (original magnification = 400k) of a thinner section (about 50 nm thick). The diameter of the hydroxyapatite crystal was 40 nm in this section. The whole set of photomicrographs are published electronically as Appendix Figure 1 (http://jdr.sagepub.com/supplemental).

produce a clean enamel surface free of debris (Uncut group). The ultra-mild one-step self-etch adhesive Clearfil S^3 Bond (Kuraray Medical, Tokyo, Japan) was applied according to the manufacturer's instructions, followed by a thin layer of flowable composite (Clearfil Protect Liner F, Kuraray Medical). Light-curing was performed by an Optilux 500 (Demetron/Kerr, Danbury, CT, USA) device with a light output not less than 600 mW/cm^2. In addition, the same adhesive was applied after enamel, prepared following the 3 methods explained before, and was etched with 40% phosphoric acid (K-etchant, Kuraray Medical) for 15 sec. After bonding procedures, specimens were stored for 1 day in tap water at 37°C.

The specimens were processed according to the procedure described in detail by Van Meerbeek *et al.* (1998). Non-demineralized ultra-thin sections were cut (Ultracut UCT, Leica, Vienna, Austria) and examined by transmission electron microscopy (TEM; JEM-1200EX II, JEOL, Tokyo, Japan). After initial examination of the non-demineralized state, the TEM sections were demineralized when the TEM grid was dipped for 5 sec in a 0.1 N HCl solution, followed by careful rinsing with distilled water to remove all dissolved mineral components (Hannig *et al.*, 2002); samples were again observed by TEM.

RESULTS

TEM revealed that the cut angle of enamel was tilted in a particular direction for each specimen, resulting in a widely varying morphology, crystal orientation, and the typical 'key-hole' configuration. A thinner section revealed near-homogeneous shearing of enamel crystals, and at the highest magnification of 400,000x, the mean size of the enamel crystals was about 40 nm (Fig. 1).

TEM of non-demineralized sections revealed clear differences in substrate roughness and smear-layer thickness for the different surface-preparation methods applied. Lab-demineralization of sections clearly disclosed that the different smear layers also resulted in different resin-infiltration patterns. Uncut enamel specimens showed no smear layer (Fig. 2a), but a thin aprismatic enamel layer of about 100 nm (Fig. 2b). SiC preparation resulted in thin and flat surfaces consisting of fragmented hydroxyapatite crystals (Fig. 3a). By the shearing/pushing motion during grinding, these small crystals were arranged in a very compact way (Fig. 3b). Bur preparation, in contrast, did increase surface roughness, and a thicker layer of debris was deposited on the surface, along with some loosely attached enamel particles (Fig. 4a). Furthermore, relatively large subsurface cracks, filled with adhesive resin, were observed (Fig. 4b).

Despite its acidity (pH ≈ 2.7), the adhesive was unable to dissolve the SiC-produced or the bur-prepared smear-layer (Figs. 3b, 4b), but impregnated and encapsulated the smear debris to a certain extent. Within this 'resin-smear complex', hydroxyapatite, often fragmented, was abundantly present. Resin infiltration progressed preferentially along cracks and voids between crystallites and along the inter-crystallite space. Therefore, the pattern of resin-infiltration differed, depending on how the surface was prepared. To uncut enamel, resin-impregnation was mostly limited to a depth ranging from nearly zero up to 400 nm, although mostly the infiltration did not extend beyond the aprismatic enamel layer (about 100 nm) (Fig. 2b). A much deeper resin infiltration was observed for the SiC-prepared specimens. The adhesive resin effectively impregnated in between all fragmented hydroxyapatite crystals. This resulted in a fine reticular maze, enveloping most crystals individually. The infiltration depth varied widely with the region, from about 500 nm up to more than 1.5 µm. Also, for bur-prepared specimens, the inter-crystallite resin network extended relatively deep into enamel (up to 500 nm). In contrast to SiC-prepared enamel, resin impregnation was not uniform, but occurred mostly along cracks and voids. From these voids, the deeper unaffected enamel was infiltrated for a maximum of a few hundred of nanometers, so that the 'resin-smear complex' contained many areas that were not infiltrated by resin.

Prior phosphoric-acid-etching revealed no ultrastructural differences for the different surface-preparation methods applied. The aprismatic layer and resin-smear complex were completely removed, while the sizes of the enamel crystals at the interface

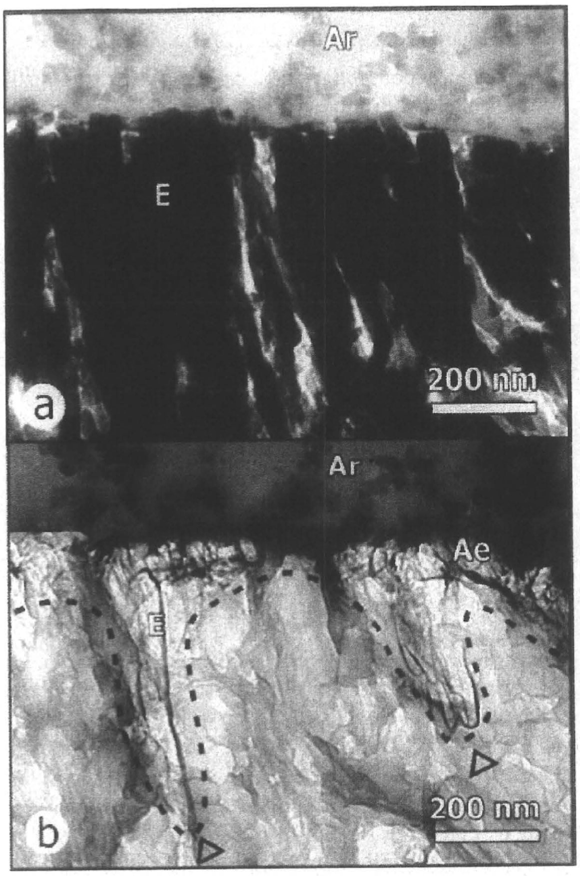

Figure 2. An 'ultra-mild' self-etch adhesive bonded to uncut enamel. **(a)** A tight, void-free interface between the adhesive resin and the enamel surface can be observed. Even at this high magnification (original magnification = 100k), no morphologic features of interaction or demineralization were revealed in this non-decalcified section. **(b)** Corresponding decalcified section of (a) at exactly the same spot, revealing 2 types of resin infiltration: one closely related to the crystal structure and mostly limited to the aprismatic enamel layer (mainly less than 100 nm), while the other appears more related to small defects in the crystal structure, so that the resin could infiltrate up to 500 nm deep (open arrows). This infiltration occurred every few hundred nanometers, suggesting facilitated infiltration among some enamel crystals. Black dotted line = bottom of inter-crystal network. Ae = Aprismatic enamel; Ar = Adhesive resin; E = Enamel. The whole set of photomicrographs are published electronically as Appendix Figure 2 (http://jdr.sagepub.com/supplemental).

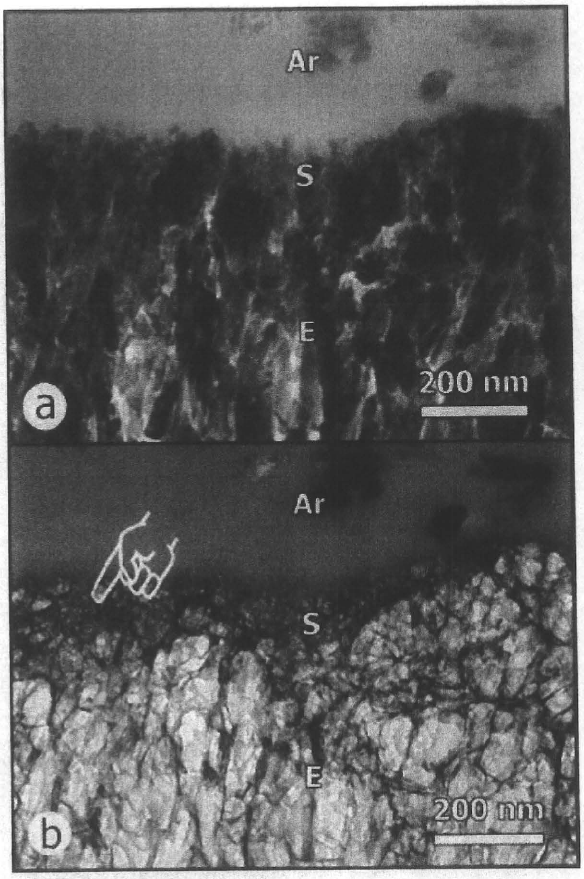

Figure 3. An 'ultra-mild' self-etch adhesive bonded to SiC-ground enamel. **(a)** A tight, void-free interface between the adhesive resin and the enamel surface can be observed. At the surface, numerous small (less than 20 nm) hydroxyapatite crystals represent smear particles. **(b)** Corresponding decalcified section of (a), in which no aprismatic layer was disclosed. The resin-smear complex and inter-crystal resin network are revealed by this demineralization step. The resin appeared to have easily penetrated the fragmented crystals at the surface (hand pointer). But also deeper, the adhesive resin seemed to have penetrated the enamel quite uniformly, thereby enveloping individual hydroxyapatite crystals and leaving a fine reticular resin network in this decalcified section. The crystal-resin interface appears as a thin black line delimiting the demineralized crystal. This feature might be related to the more acid-resistant calcium-MDP salt, as opposed to the hydroxyapatite core of the crystal. Ar = Adhesive resin; E = Enamel; S = Smear. The whole set of photomicrographs are published electronically as Appendix Figure 3 (http://jdr.sagepub.com/supplemental).

were clearly decreased by partial dissolution through phosphoric acid. The interface between rod and interrod enamel was selectively dissolved in all groups. The only difference observed among the 3 preparation methods was that the inter-crystallite resin network of uncut enamel was deeper than that of both the prepared substrates.

DISCUSSION

As the hardest connective tissue in the human body, dental enamel consists of 96wt% mineral, 4wt% organic material, and water. Previous studies have disclosed some of the unique microstructure of enamel (Nanci, 2008). The principal structure of enamel is based on nano-sized fibril-like hexagonal hydroxyapatite crystals, which are further organized into groups (enamel rod) (Farina *et al.*, 1999). Cui and Ge (2007) investigated a hierarchical assembly of enamel structure using various microscopic explorations. The shape of an enamel rod in cross-section is apparently un-deformed, and the typical keyhole-like aspect only suggests that the cut angle was tilted in some particular direction. The shape of the enamel rod in cross-section depends on the cut angle relative to the long axis of the prism/interprism continua (enamel rod). It varies gradually from circular (when cut perpendicularly) to rectangular (when cut parallel) (Eisenmann,

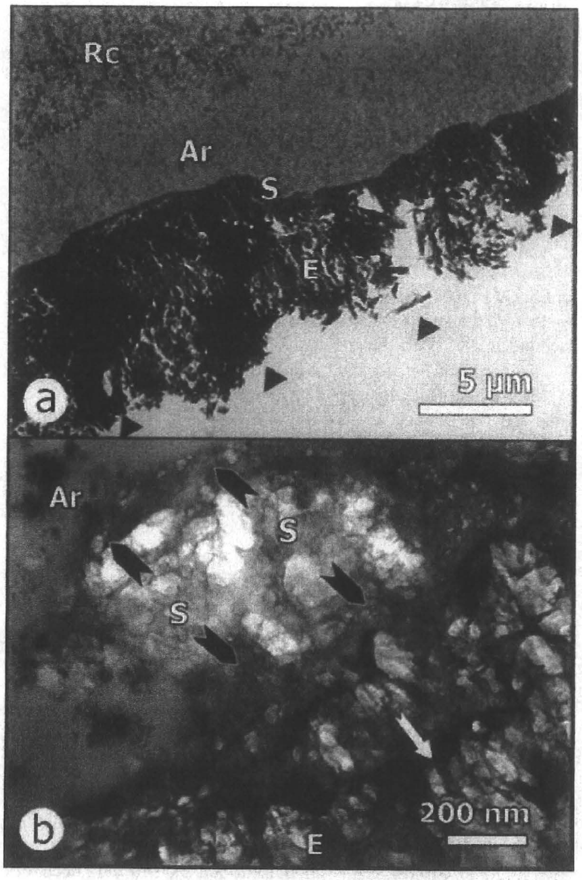

Figure 4. An 'ultra-mild' self-etch adhesive bonded to bur-cut enamel. **(a)** Overview of the interface. The medium-grit diamond bur exposed a clearly irregular surface. The smear layer varied largely in thickness. Note the repetitive changes in crystal density at about every 5 µm, consistent with the sizes of the enamel rods (black arrowheads). The bur-cut surface irregularity appears unaffected by the difference in crystal density. **(b)** Decalcified section of the interface at a higher magnification. A thicker and larger, but more irregular, smear layer is deposited on enamel (delimited by black arrows), as compared with that of SiC-ground enamel. A fine inter-crystallite network, including subsurface cracks, was observed (white arrow = subsurface cracks filled with adhesive resin). The inner part of the enamel crystal is clearly less electron-dense, suggesting poor resin-infiltration, while the outer part was better stabilized by resin and connected to the underlying enamel. Ar = Adhesive resin; E = Enamel; Rc = Resin composite; S = Smear. The whole set of photomicrographs are published electronically as Appendix Figure 4 (http://jdr.sagepub.com/supplemental).

1994). The typical diameter of a human enamel rod has been reported to vary between 5 and 7 µm (Ge *et al.*, 2005). In the present study, enamel rods and crystals were clearly observed.

Inter- and intra-crystallite demineralization and subsequent resin infiltration into enamel are more difficult to assess than resin hybridization of dentin, even by high-resolution TEM. A viable method to disclose inter-crystallite nano-retention is by decalcifying the TEM section with a HCl solution (Hannig *et al.*, 2002). In the present study, 3 types of enamel surface-preparation methods were used and ultra-morphologically characterized after treatment with an ultra-mild one-step self-etch adhesive (Clearfil S^3 Bond, pH ≈ 2.7). TEM evaluation is the method of choice for ultra-morphological comparison of these surface-preparation methods, since the combination of resolution and image detail is unsurpassed. The inter-crystal resin network revealed by TEM has previously been considered as a resin-infiltrated enamel inter-diffusion zone or enamel hybrid layer (Nakabayashi and Pashley, 1998; Pashley and Tay, 2001). The composition of this inter-crystallite network is much more variable than that of the hybrid layer typically produced at dentin. In some areas, resin hardly impregnated the surface, while in other areas the network extended to a depth of over 1.5 µm.

Clearfil S^3 Bond is an ultra-mild self-etch adhesive. The interaction with dentin has been described in detail (Koshiro *et al.*, 2006). Even to this less-mineralized dentin substrate, only a 'nano-interaction zone' is produced without distinct demineralization. Since enamel contains even more hydroxyapatite than dentin, it is probable that more hydrogen ions (H^+) released from the adhesive are neutralized by the enamel smear, even before the intact surface is reached. This was corroborated by TEM in this study, since in none of the sections was surface demineralization observed, regardless of the preparation method applied. Hence, micro-mechanical interlocking of the adhesive resin into the surface is more dependent on the surface receptiveness, which differed for the 3 surface-preparation methods in our study.

At uncut enamel, a thin aprismatic layer was observed. Even though a void-free interface was observed, this layer acted as a barrier, and in most areas the adhesive could not infiltrate beyond it. The areas that were infiltrated more deeply appeared to be related to defects in the enamel structure. SiC preparation removed the aprismatic layer. By the shearing/pushing motion, many hydroxyapatite crystals were fragmented and must have been detached from the surface. However, due to the sandpaper rubbing, these particles were compacted in the surface voids, so that a rather flat and smooth surface was obtained. Below this top layer, the enamel structure must have been altered to a certain degree, since the adhesive was able to incorporate every single crystal, resulting in a fine reticular resin network. It appears that the enamel structure was damaged, so that the inter-crystal structure was lost and consecutively infiltrated by the adhesive resin. This deep infiltration is probably more beneficial than harmful, since it facilitated a firmer micro-mechanical interlock and probably provided plenty of opportunity for the adhesive resin to interact chemically with hydroxyapatite (Yoshida *et al.*, 2004). Bur preparation, in contrast, resulted in a rougher surface. It appeared as if the diamond bur chopped off relatively big chunks of enamel, leaving an irregular substrate, while in some instances, these enamel particles remained (loosely) attached to the enamel surface. This process, however, did not affect the enamel structure itself on a micro-scale, as did the SiC-grinding. However, numerous subsurface cracks were observed. These cracks and voids served as infiltration 'highways', along which the adhesive resin formed a fine inter-crystallite resin network. Even though it did not consist of a fine reticular mesh enveloping all crystals in the enamel 'hybrid layer' (*e.g.*, in case enamel was prepared with SiC-paper), this resin network must have stabilized the enamel bond to some extent. This is corroborated by the fact that another mild self-etch adhesive (Clearfil Protect Bond, Kuraray Medical) bonded less effectively to unground than to bur-cut enamel (Ermis *et al.*, 2007).

Since the demineralization capability of ultra-mild self-etch adhesives is limited, they are more sensitive to the basic nature of the smear-layer. For example, the adhesive performed significantly better when an extra-fine diamond bur (15 μm grit-size, corresponding more to that of the sandpaper used in our study) was used, as opposed to a more commonly used regular bur (100 μm grit-size, similar to the bur used in this study) (Ermis *et al.*, 2007). This is perfectly in line with our results, where the adhesive resin was able to infiltrate the SiC-ground surface more effectively and deeply than the bur-prepared surface. In most studies investigating enamel-bonding effectiveness, SiC-ground surfaces are used, because of the ease of preparation and standardization (Amano *et al.*, 2006; Ando *et al.*, 2008; Watanabe *et al.*, 2008). This may, in part, also be due to the technical difficulties of measuring bond strength to convex, unground enamel surfaces (Kanemura *et al.*, 1999). However, our study clearly showed that SiC preparation resulted in a clearly different substrate than diamond-bur preparation, and that this must affect the resultant bonding effectiveness.

Even though it is not recommended by the manufacturer, in many clinical situations, this ultra-mild self-etch adhesive is applied to unground enamel, *e.g.*, when composite is applied beyond the cavity margins, when orthodontic brackets are bonded, or when pits and fissures are sealed. In these cases, the bonding effectiveness is probably low, and additional micro-mechanical interlocking, as can be obtained with prior phosphoric-acid-etching, is highly recommended (Watanabe *et al.*, 2008). TEM of the different phosphoric-acid-etched enamel surfaces revealed no differences in surface roughness. The acid effectively removed the smear complex, the aprismatic layer, and the cracks prepared during preparation. Hence, phosphoric-acid-etching is effective to rule out preparation side-effects and ensures a deep and uniform substrate infiltration. The only difference observed was that the use of phosphoric acid on unprepared specimens resulted in a somewhat deeper infiltration. This may, however, be related to the differences in local micro-morphology and/or enamel rod angulations between surface and subsurface enamel.

It is concluded that the surface-preparation method significantly affects the nature of the smear layer and thus the interaction of, in particular, ultra-mild self-etch adhesives. Adhesion to unprepared enamel appeared most challenging. Smear-layer removal by phosphoric-acid-etching and polishing can improve adhesion to diamond-bur-prepared enamel surfaces. In light of the increased popularity of (ultra-)mild self-etch adhesives, adhesion to enamel requires more attention regarding surface-preparation methods than more old-fashioned etch and rinse adhesives.

ACKNOWLEDGMENTS

This study was supported by the FWO grant G.0206.07 and the K.U. Leuven grant OT/06/55. A. Mine has been granted a scholarship of the Government of Flanders for the academic year 2006-2007 to conduct research at the Leuven BIOMAT Research Cluster of the Catholic University of Leuven. K.L. Van Landuyt has been appointed as Post-doctoral Research Fellow for Scientific Research of Flanders.

REFERENCES

Amano S, Yamamoto A, Tsubota K, Rikuta A, Miyazaki M, Platt JA, *et al.* (2006). Effect of thermal cycling on enamel bond strength of single-step self-etch systems. *Oper Dent* 31:616-622.

Ando S, Watanabe T, Tsubota K, Yoshida T, Irokawa A, Takamizawa T (2008). Effect of adhesive application methods on bond strength to bovine enamel. *J Oral Sci* 50:181-186.

Buonocore MG (1955). A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. *J Dent Res* 34:849-853.

Buonocore MG, Matsui A, Gwinnett AJ (1968). Penetration of resin dental materials into enamel surfaces with reference to bonding. *Arch Oral Biol* 13:61-70.

Cui FZ, Ge J (2007). New observations of the hierarchical structure of human enamel, from nanoscale to microscale. *J Tissue Eng Regen Med* 1:185-191.

Eisenmman DR (1994). Enamel structure. In: *Oral histology: development, structure and function*. 4th ed. Ten Cate AR, editor. St. Louis, MO: Mosby-Year Book Inc., pp. 239-256.

Ermis RB, De Munck J, Cardoso MV, Coutinho E, Van Landuyt KL, Poitevin A, *et al.* (2007). Bonding to ground versus unground enamel in fluorosed teeth. *Dent Mater* 23:1250-1255.

Farina M, Schemmel A, Weissmüller G, Cruz R, Kachar B, Bisch PM (1999). Atomic force microscopy study of tooth surfaces. *J Struct Biol* 125:39-49.

Ge J, Cui FZ, Wang XM, Feng HL (2005). Property variations in the prism and the organic sheath within enamel by nanoindentation. *Biomaterials* 26:3333-3339.

Hannig M, Bock H, Bott B, Hoth-Hannig W (2002). Inter-crystallite nanoretention of self-etching adhesives at enamel imaged by transmission electron microscopy. *Eur J Oral Sci* 110:464-470.

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

Koshiro K, Sidhu SK, Inoue S, Ikeda T, Sano H (2006). New concept of resin-dentin interfacial adhesion: the nanointeraction zone. *J Biomed Mater Res Part B: Appl Biomater* 77:401-408.

Nakabayashi N, Pashley DH (1998). *Hybridization of dental hard tissues.* Tokyo, Japan: Quintessence Publishing Inc.

Nanci A (2008). Enamel: composition, formation and structure. In: *Ten Cate's oral histology: development, structure, and function*. 7th ed. Ten Cate AR, editor. St. Louis, MO: Mosby Inc. (Elsevier Health Science), pp. 141-190.

Nishiyama N, Tay FR, Fujita K, Pashley DH, Ikemura K, Hiraishi N, *et al.* (2006). Hydrolysis of functional monomers in a single-bottle self-etching primer—correlation of 13C NMR and TEM findings. *J Dent Res* 85:422-426.

Pashley DH, Tay FR (2001). Aggressiveness of contemporary self-etching adhesives. Part II: etching effects on unground enamel. *Dent Mater* 17:430-444.

Peumans M, De Munck J, Van Landuyt K, Lambrechts P, Van Meerbeek B (2007). Five-year clinical effectiveness of a two-step self-etching adhesive. *J Adhes Dent* 9:7-10.

Salz U, Zimmermann J, Zeuner F, Moszner N (2005). Hydrolytic stability of self-etching adhesive systems. *J Adhes Dent* 7:107-116.

Sarr M, Kane AW, Vreven J, Mine A, Van Landuyt KL, Peumans M, *et al.* (2009). Microtensile bond strength and interfacial characterization of 11 contemporary adhesives bonded to bur-cut dentin. *Oper Dent* 35: 94-104.

Van Meerbeek B, Yoshida Y, Lambrechts P, Vanherle G, Duke ES, Eick JD, *et al.* (1998). A TEM study of two water-based adhesive systems bonded to dry and wet dentin. *J Dent Res* 77:50-59.

Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, *et al.* (2003). Buonocore memorial lecture. Adhesion to enamel and dentin: current status and future challenges. *Oper Dent* 28:215-235.

Watanabe T, Tsubota K, Takamizawa T, Kurokawa H, Rikuta A, Ando S, *et al.* (2008). Effect of prior acid etching on bonding durability of single-step adhesives. *Oper Dent* 33:426-433.

Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, *et al.* (2004). Comparative study on adhesive performance of functional monomers. *J Dent Res* 83:454-458.

A 15-year Clinical Comparative Study of the Cumulative Survival Rate of Cast Metal Core and Resin Core Restorations Luted with Adhesive Resin Cement

Takayuki Hikasa, DDS[a]/Yoshizo Matsuka, DDS, PhD[b]/Atsushi Mine, DDS, PhD[c]/
Hajime Minakuchi, DDS, PhD[d]/Emilio Satoshi Hara, DDS[a]/Bart Van Meerbeek, DDS, PhD[e]/
Hirofumi Yatani, DDS, PhD[f]/Takuo Kuboki, DDS, PhD[g]

Purpose: The aim of this study was to compare the core survival rates (CSRs) of cast metal versus resin core restorations luted with adhesive resin cement, as well as to determine the risk factors for core failure. ***Materials and Methods:*** Nine hundred ninety-one patients (2,124 cores) who received either cast metal or resin cores luted with adhesive resin cement at the Fixed Prosthodontic Clinic of Okayama University Dental Hospital between April 1988 and December 1991 and whose structured clinical core record was filled appropriately comprised the study subjects. The clinical core record included information regarding patient age, sex, core restoration type, tooth location, tooth type, remaining coronal dentin, and root canal form. CSRs, as well as causes for failure, were analyzed 15 years postinsertion. Since 381 patients lacked data regarding predictors for core failure, a subsample of 610 patients (1,053 cores) was used for the subsequent risk factor analysis. ***Results:*** The cumulative CSR of resin cores (78.7%) was significantly higher than that of cast metal cores (55.4%; log-rank test, $P < .0001$). The Cox proportional hazards test revealed that sex (male, $P < .0001$), absence of remaining coronal dentin ($P = .0057$), core restoration type (cast metal, $P = .0186$), and higher age at core insertion ($P = .0380$) were significant predictors for core failure. The incidence of complications, such as core loosening ($P = .0016$) and tooth extraction ($P < .0001$), was significantly higher in cast metal cores. ***Conclusions:*** Cast metal cores were associated with a significantly lower CSR than resin cores, and significant risk factors for core failure were sex (male), absence of remaining coronal dentin, core restoration type (cast metal), and higher age at core insertion. *Int J Prosthodont 2010;23:397–405.*

Endodontically treated teeth often display decreased physical resistance due to considerable tooth structure damage caused by caries, consecutive filling failures, or fractures.[1,2] In severe cases resulting from a large amount of tooth destruction, post and core buildups have been used prior to the definitive restoration.[1–4] Two different buildup techniques are widely accepted, namely the indirect cast metal post and core restoration (cast metal core) and the direct prefabricated post and resin core restoration (resin core).[1–7]

In daily clinical decision making, knowledge of the long-term core success rates associated with different techniques, as well as being aware of the risk factors for failure, are essential for core selection. Although there are numerous published studies analyzing core survival rates (CSRs), it is still unclear which treatment is preferable for a specific tooth condition (for example, a two- or three-wall remaining coronal dentin).[1,3] Five systematic reviews resulted in an impractical study comparison because of a great diversity in cement materials, techniques, sampling, statistical methods, and study design, as well as a lack of controlled prospective studies and randomized clinical trials.[1,3–6]

---

[a] *Postgraduate Student, Oral Rehabilitation and Regenerative Medicine, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, Okayama, Japan.*

[b] *Associate Professor, Oral Rehabilitation and Regenerative Medicine, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, Okayama, Japan.*

[c] *Postdoctoral Researcher, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Leuven, Belgium.*

[d] *Assistant Professor, Oral Rehabilitation and Regenerative Medicine, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, Okayama, Japan.*

[e] *Professor, Leuven BIOMAT Research Cluster, Department of Conservative Dentistry, School of Dentistry, Oral Pathology and Maxillo-Facial Surgery, Catholic University of Leuven, Leuven, Belgium.*

[f] *Professor and Chair, Fixed Prosthodontics, Osaka University Graduate School of Dentistry, Osaka, Japan.*

[g] *Professor and Chair, Oral Rehabilitation and Regenerative Medicine, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, Okayama, Japan.*

Correspondence to: *Prof Takuo Kuboki, Oral Rehabilitation and Regenerative Medicine, Okayama University Graduate School of Medicine, Dentistry and Pharmaceutical Sciences, 2-5-1 Shikata-cho, Okayama, 700-8525, Japan. Fax: +81 86 235 6684. Email: kuboki@md.okayama-u.ac.jp*

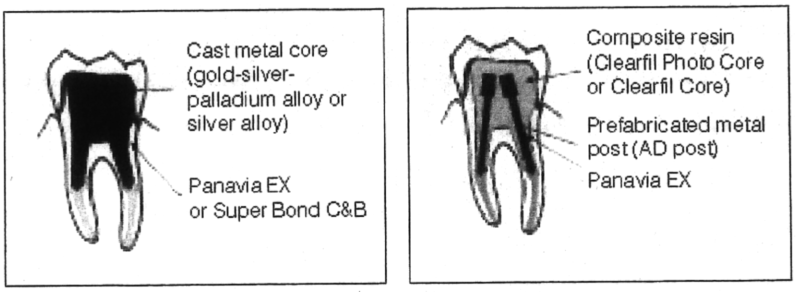

Fig 1 Core types: **(left)** indirect cast metal core and **(right)** direct resin core.

The type of cement material used has been reported to play a crucial role in increasing post retention strength and tooth fracture resistance in in vitro and clinical follow-up studies.[3,8–11] Although conventional cements (zinc phosphate or glass-ionomer cements) are widely used because of their simplicity and proven efficacy, adhesive resin cement is regarded as effective in improving the long-term success of cores, mainly because its physical properties are similar to dentin. Furthermore, adhesive resin cement has displayed a high degree of clinical reliability regarding bond adhesion to root dentin.[3,11,12]

Despite an increasing number of clinicians using adhesive resin cement for both resin and cast metal core restorations, no study has been published showing the CSRs and risk factors for failure of adhesively luted cast metal cores. In addition, most studies have reported retrospective CSRs of cast metal cores luted with conventional cements and resin cores using various types of prefabricated posts 5 or 10 years postinsertion.[1–4] Also, longitudinal follow-up studies evaluating CSRs for more than 10 years are scarce.[7]

Therefore, this clinical comparative cohort study was designed to verify the 15-year survival rate of cast metal core and resin core treatments luted with adhesive resin cements. This study also sought to identify the risk factors for core failure. The null hypothesis was that no significant difference in the CSR would be observed between the two core groups.

Materials and Methods

Study Population

The study subjects were selected among patients who consecutively attended the Fixed Prosthodontic Clinic of Okayama University Dental Hospital, Okayama, Japan, between April 1988 and December 1991. Eligible patients were those who received cast metal or resin core restorations and whose structured clinical core records were completed appropriately. A total of 1,024 subjects and 2,174 cores were eligible. Thirty-three subjects were excluded due to unmatched data in hospital chart and core records. Therefore, the actual sample for CSR analysis consisted of 991 subjects (male: 276, female: 715) and 2,124 cores (cast metal: 372, resin: 1,752).

Among the study population, 381 subjects lacked data concerning the analyzed predictor variables for core failure and were consequently excluded from the subsequent Cox proportional hazards analysis. Therefore, the subsample for the risk analysis consisted of 610 patients (male: 155, female: 455) and 1,053 cores (cast metal: 187, resin: 866).

This study protocol was reviewed and approved by the Ethical Committee for Human Study of Okayama University Graduate School of Medicine, Dentistry, and Pharmaceutical Sciences (no. 176).

Core Restoration Techniques

Figure 1 shows a schematic diagram of the two types of core restorations. For the resin core restoration, the root canal was drilled parallel-walled with a Largo Peeso Reamer (Dentsply), and a prefabricated metal post (AD post, Kuraray) of an identical diameter was cemented with Panavia EX (Kuraray), according to the manufacturer's instructions. The bonding agent was either Clearfil New Bond (Kuraray) or Clearfil Photo Bond (Kuraray) after dentin pretreatment with 40% phosphoric acid (30 seconds) and 10% sodium hypochlorite (60 seconds).[13–16] After post cementation, composite resin (Clearfil Photo Core or Clearfil Core, Kuraray) was used for the core buildup.

For the cast metal core preparation, the root canal cavity was drilled with a taper reamer (Dentech) and an impression was made using a silicone impression material (Exafine, GC). The cast metal core was made of a gold-silver-palladium alloy (Castwell M.C. [12% gold], GC) or a silver alloy (Miro Bright, GC) and was prepared by one dental laboratory. The cast metal core was luted with either Panavia EX or Super Bond C&B (Sun Medical) adhesive cement. When the core was cemented with Panavia EX, the aforementioned steps used for resin core cementation were performed. For cores luted with Super Bond C&B, dentin pretreatment was performed with 10% citric acid and 3% ferric chloride for 10 seconds, according to the manufacturer's instructions.

The selection of the adhesive cements and core materials was determined according to the individual preferences of 45 experienced clinicians (residents, postgraduate students, and faculty professors) who treated the patients. Immediately after core insertion, the treating clinician filled out the hospital chart and other clinicians completed the clinical core record according to their own inspection. The type of restoration placed on the treated tooth was either a full crown or a fixed partial denture.

Classification of Survival, Failure, and Censored

The core condition (survival, failure, or censored) was assessed by one investigator from data contained in the hospital chart and the clinical core record. Surviving cores included cores that remained in their original form up to the analysis endpoint (November 16, 2004). Failures involved one of the following three complications: core loosening (due to caries, root/tooth fracture, or post fracture), core removal (due to caries, root/tooth fracture, or periapical periodontitis), or tooth extraction or hemisection (due to caries, root/tooth fracture, post fracture, marginal periodontitis, periapical periodontitis, or an unspecified reason). Censored cases involved cores in their original form for which clinical follow-up was lost prior to the endpoint due to the patient's withdrawal from the dental hospital (unwilling to return, change of address, or change of treatment to another dental office) or the patient's death (alternative outcome).

In instances of restorations involving multiple teeth (eg, fixed partial dentures), each tooth was considered as one individual core.

Risk Factors for Core Failure and Complications

Baseline data of risk factors included the core restoration type (cast metal or resin core), age at core insertion, sex, date of core insertion (working duration), remaining coronal dentin (absent or present), core margin location (below or above crown margin), root canal forms (straight or funnel), tooth location (mandible or maxilla), tooth type (anterior or posterior), and index of decayed, missing, and filled teeth (DMFT Index).

The DMFT Index was followed-up by the periodic recall of patients. The number of missing teeth was counted to show the overall intraoral condition in both core groups throughout the entire study period.

Statistical Analysis

The chi-square test and t test were used to compare the baseline data between the cast metal and resin core groups of the actual sample for CSR estimation, as well as the subsample for the risk factor analysis for core failure.

The CSR was calculated by the Kaplan-Meier method and lifetable analysis.[17] The log-rank test was used to compare the survival curves between the two core groups.[18] Comparative analyses between the two adhesive cements used in the cast metal core group were performed with Kaplan-Meier and log-rank tests. Log-rank analysis was also used to verify whether a significant difference between core cementation performed by faculty professors or postgraduate students and residents would account for CSR differences.

Finally, the Cox proportional hazards test was used to calculate the relative risk for core failure of each predictor variable. Intergroup comparison of core complications was analyzed using the chi-square test. The significance level was set at $P < .05$.

Results

Baseline Data Comparison

A baseline comparison between the cast metal and resin core groups of the intended and actual samples was performed based on age at core insertion, sex, DMFT Index, and number of remaining teeth (Table 1). There was no statistical difference in regard to the mean age at core insertion (cast metal: 52.0 ± 14.2 years, resin: 50.6 ± 13.8 years) and sex ratio (male: 51, female: 146 for the cast metal core; male: 225, female: 569 for the resin core) between the intended and actual samples or between the two core groups. In addition, there was no significant difference in the remaining number of teeth between the two core groups throughout the entire study period (Fig 2).

However, an intergroup baseline data comparison of the subsample for subsequent analysis of risk factors for core failure revealed significant differences in regard to remaining coronal dentin ($P < .0001$), core margin location ($P < .0001$), root canal form ($P < .0001$), tooth location ($P < .0001$), tooth type ($P = .0001$), and DMFT Index ($P = .0001$) among the two core groups (Table 2).

CSR and Risk Factors for Core Failure

A Kaplan-Meier analysis indicated that the 15-year estimated cumulative survival rate of the resin core (78.7%) was significantly higher than that of the cast metal core (55.4%; log-rank test, $P < .0001$) (Fig 3). A lifetable analysis of survival, failure, and censored cores is shown in Table 3.

No significant difference was observed in CSRs between the two resin cements (Panavia EX and Super Bond C&B) used in the cast metal core group (log-rank

Table 1 Baseline Data Comparison Between Cast Metal and Resin Core Groups of the Intended and Actual Study Samples

	Cast metal core			Resin core		
	Intended sample	Actual sample	*P*	Intended sample	Actual sample	*P*
Patients (cores)	220 (407)	197 (372)		804 (1,767)	794 (1,752)	
Age at core installation (mean ± SD) (y)	51.9 ± 14.2[a]	52.0 ± 14.2[b]	.9965[†]	50.6 ± 13.8[a]	50.6 ± 13.8[b]	.9954[†]
Age range (y)	16–86	16–78		16–82	16–82	
Sex (M/F)	52/168[c]	51/146[d]	.9705[‡]	227/577[c]	225/569[d]	.6587[‡]
DMFT index (mean ± SD)		19.8 ± 5.4[e]			18.0 ± 6.1[e]	
No. of remaining teeth (mean ± SD)		22.8 ± 5.2[f]			23.4 ± 5.0[f]	

SD = standard deviation; M = male; F = female.
[a]*P* = .0793[†]; [b]*P* = .0927[†]; [c]*P* = .1366[‡]; [d]*P* = .3608[‡]; [e]*P* = .9877[†]; [f]*P* = .1775.[†]
[†]*t* test; [‡]chi-square test.

Table 2 Baseline Intergroup Comparison of the Subsample for Analysis of the Risk Factors for Core Failure

	Cast metal core	Resin core	*P*
Patients (cores)	111 (187)	499 (866)	
Age at core installation (mean ± SD) (y)	50.6 ± 15.1	50.5 ± 13.6	.9389[*]
Age range (y)	18–76	16–82	
Sex (M/F)	23/88	132/367	.2096[†]
Remaining coronal dentin (absent/present)	129/58	160/706	< .0001[†]
Core margin location (below crown margin/normal)	62/125	129/737	< .0001[†]
Root canal form (straight/funnel)	126/61	780/86	< .0001[†]
Tooth location (mandible/maxilla)	135/52	485/381	< .0001[†]
Tooth type (anterior/posterior)	85/102	268/598	.0001[†]
DMFT index (mean ± SD)	21.1 ± 5.5	18.9 ± 5.7	.0001[*]
No. of remaining teeth (mean ± SD)	23.5 ± 4.8	23.8 ± 4.6	.3883[*]

SD = standard deviation; M = male; F = female.
[*]*t* test; [†]chi-square test.

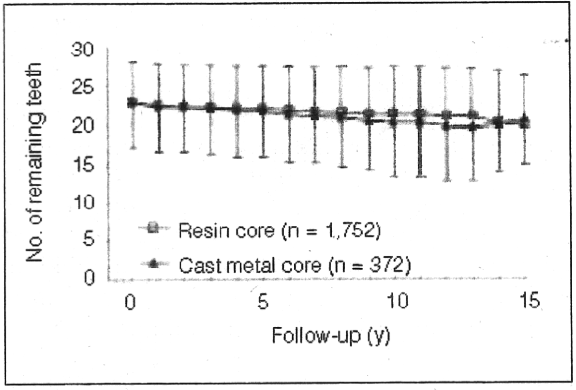

Fig 2 Comparison of the number of remaining teeth between cast metal and resin core groups in the actual study sample. A *t* test revealed no statistical difference at any point in time.

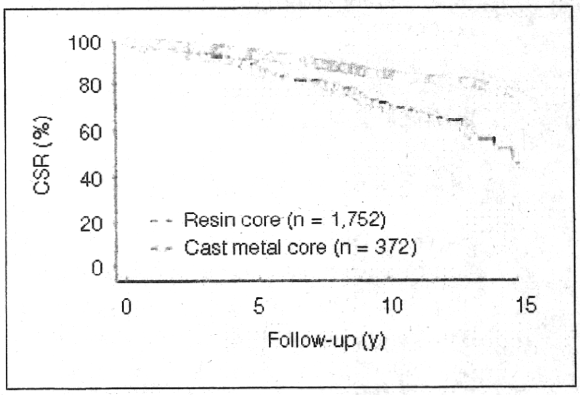

Fig 3 Kaplan-Meier 15-year survival curves of cast metal cores (55.4%) and resin cores (78.7%). The significant statistical difference was verified by the log-rank test (*P* < .0001).

test, *P* = .2921) (Fig 4). As a result, they were recognized to be a homogenous group in the subsequent statistical analyses.

Moreover, the log-rank test showed no significant difference in CSRs between the cores cemented by faculty professors or by postgraduate students and residents, neither in the entire core sample analysis (*P* = .2120) nor in each of the core groups (cast metal core: *P* = .4430, resin core: *P* = .4782).

According to the Cox proportional hazards test, sex (male, *P* < .0001), absence of remaining coronal dentin (*P* = .0057), core restoration type (cast metal core, *P* = .0186), and aging (*P* = .0380) were significant risk factors for core failure (Table 4).

Table 3 Lifetable of Survival, Failure, and Censored Cores

Year	Cast metal core				Resin core			
	Survival (n)	Censored (n)	Failure (n)	Survival ratio (%)	Survival (n)	Censored (n)	Failure (n)	Survival ratio (%)
0–1	372	79	10	100.0	1,752	302	12	100.0
1–2	283	51	3	97.0	1,438	152	17	99.3
2–3	229	24	5	95.9	1,269	94	13	98.0
3–4	200	15	2	93.7	1,162	125	17	97.0
4–5	183	8	6	92.7	1,020	77	13	95.5
5–6	169	6	10	89.6	930	52	16	94.2
6–7	153	11	3	84.2	862	44	10	92.5
7–8	139	9	4	82.5	808	54	23	91.4
8–9	126	6	7	80.0	731	50	17	88.7
9–10	113	3	6	75.5	664	61	13	86.6
10–11	104	13	4	71.4	590	37	13	84.8
11–12	87	12	4	68.5	540	38	8	82.9
12–13	71	20	2	65.1	494	53	2	81.6
13–14	49	31	4	63.0	439	179	11	81.3
14–15	14	7	2	55.4	249	124	6	78.7

Fig 4 Kaplan-Meier survival curves of Panavia EX and Super Bond C&B used for cast metal core cementation. There was no statistical difference between the two cements (log-rank test, *P* = .2921).

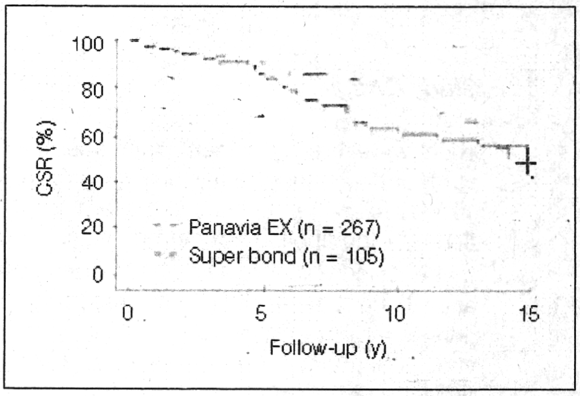

Table 4 Multiple Regression Analysis of Risk Factors for Failure of Core Restorations

Predictor variables	*P**	95% CI	Relative risk
Sex (female or male)	< .0001	0.282–0.598	0.411
Remaining coronal dentin (absent or present)	.0057	1.192–2.806	1.828
Type of core restoration (cast metal or resin)	.0186	1.096–2.730	1.729
Age at core installation (y)	.0380	1.001–1.034	1.017
Tooth location (mandible or maxilla)	.3358	0.824–1.765	1.206
Core margin location (below crown margin or normal)	.4041	0.762–1.966	1.224
Tooth type (anterior or posterior)	.7486	0.714–1.598	1.068
DMFT Index	.8507	0.971–1.036	1.003
Root canal form (straight or funnel)	.8712	0.572–1.604	0.958

CI = confidence interval.
*Cox proportional hazards test.

The failure rates were 19.4% and 11.0% for cast metal and resin cores, respectively (Table 5). The major core complication was tooth extraction/hemisection (cast metal: 14.0%, resin: 7.2%), followed by core loosening (cast metal: 3.8%, resin: 1.4%). The causes of tooth extraction/hemisection included caries (cast metal: 4.8%, resin: 1.2%), tooth fracture (cast metal: 2.4%, resin: 0.7%), and marginal periodontitis (cast metal: 2.4%, resin: 1.0%), while caries deterioration (cast metal: 3.5%, resin: 1.0%) was the main factor causing core loosening (Table 5).