Environmental observation of enamel crack and resin-tooth cavity gap formation

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Purpose: The aim of this study was to observe marginal sealing and resin composite adaptation to cavity walls using the dye penetration test and environmental scanning electron microscope.
Materials and Methods: Cylindrical cavities were prepared on cervical regions of teeth. Teeth were restored with Clearfil Liner Bond 2V adhesive and filled with Clearfil Photo Bright or Palfique Estelite resin composites. These resin composites were cured with a conventional light-curing method. After thermal cycling, the specimens were subjected to the dye penetration test to evaluate marginal sealing and resin composites adaptation to the cavity walls. These resin-tooth interfaces were then observed using environmental scanning electron microscope.
Results: Enamel crack formation was observed in all specimens. Gap formation between the resin composite and cavity walls was observed for both resin composites.
Conclusion: Environmental scanning electron microscope showed high enamel crack formation for both resin composites after light curing. Enamel cracks were located at 25-100 μm from the resin-enamel interface on environmental scanning electron microscope observations. Enamel cracks were observed in the enamel surface to the dentin-enamel junction. Environmental scanning electron microscope showed cavity wall gap formation in the dentin-adhesive interface or resin-adhesive interface.

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Key Words: adaptation, dye penetration test, environmental SEM, polymerization, resin composite

Introduction

Adhesive dentistry is an important field of dental practice, particularly because of recent increases in the cases of abrasion and erosion due to an aging society, eating disorders, and reflux esophagitis. Teeth with abrasions and erosion cannot be treated for conventional Black's cavities with metal inlays; they can only be restored with resin composites using a direct bonding technique.

However, resin composite polymerization results in volumetric shrinkage, also known as polymerization shrinkage, and the ensuing stress created results in greater gap formation between the resin and cavity wall.[1-4] These marginal gaps and subsequent microleakage may cause marginal staining, postoperative sensitivity,[5,6] and secondary caries. In addition, gap formation between the restoration and cavity wall may result in pain on biting and adhesion failure after repeated occlusal loading. Light-cured resin composites are widely used in clinical practice because of their esthetic advantages, ease of use, good bonding to tooth structures, and excellent mechanical properties.

The polymerization reaction for light-cured composites is rapid, which results in the development of higher stresses in cured materials than in self-activated materials.[7] Furthermore, the maximum interfacial stress generated at cavity walls filled with light-cured composites is twice compared with that generated with self-cured composites.[8] Therefore, gap formation between the resin and cavity wall because this stress is greater when light-cured materials are used than when self-cured materials are used. In addition, when the bond strength exceeds these polymerization shrinkage stresses, a crack is initiated in the tooth structure, generally in the enamel,[4,9-12] resulting in the direct contact of the restoration with the oral cavity.

It is often difficult to observe real enamel cracks without damage because the enamel prisms are very fragile.[13] Observations with scanning electron microscope (SEM) require absolutely dry specimens. Therefore, enamel cracks are usually observed with a stereomicroscope[10,14,15] and replica-SEM.[16,17] However, stereomicroscopic observations and impressions to create a replica also require dry specimen surfaces. These dry conditions promote enamel crack formation. In addition, the observation of the resin-dentin interface is more difficult than that of the resin-enamel interface.

Because dentin comprises a significant amount of water, it is more sensitive to environmental conditions and shrinks under dry conditions. Cryo-SEM observations do not require dry conditions. However, cryo-SEM observations require complicated handling and time-consuming specimen preparation.[18] Alternatively, environmental SEM observations can be used to observe liquid and hydrated specimens, such as native plants and living insects. In these cases, to maintain water in its liquid phase, a minimum water vapor pressure of 609 Pa is required at 0°C, and this pressure can be arbitrarily chosen for the required indication. Therefore, environmental SEM observations require a lower pressure compared with conventional SEM observations, at least in the terms of the pressure required to observe distilled water. However, environmental SEM equipment is very expensive and the observation technique is time consuming. In comparison, the dye penetration test is an easy, rapid technique to observe resin composite adaptation to the cavity wall. We observed resin-tooth interfaces by environmental SEM and the dye penetration test.

Materials and Methods

Specimen preparation

The materials, components, manufacturers, batch numbers, and bonding procedures used in the present study are listed in Table 1.

Table 1. Study materials

Material/Manufacturer	Components[a]	Batch No.	Bonding Instruction[b]
Clearfil Liner Bond 2V (Kuraray Noritake Dental Co. Ltd., Tokyo, Japan)	Primer A: MDP, HEMA, dimethacrylates, photoinitiator, water, others	00147B	a, b, c, d, e, f
	Primer B: HEMA , dimethacrylates, accelerator, water	00145B	
	Bond A: MDP, HEMA, Bis-GMA, dimethacrylates, photoinitiator, microfiller, others	00238A	
Clearfil Photo Bright (Kuraray Noritake Dental Co. Ltd.) shade (US)	silanated colloidal silica, prepolymerized organic filler containing colloidal silica, Bis-GMA, dimethacrylates, photoinitiator, others Filler load: 82 wt%	00044C	
Palfique Estelite (Tokuyama Dental Co. Ltd., Tokyo, Japan) shade (A3)	silica-zirconia filler, Bis-GMA, TEGDMA, photo initiator Filler load: 82 wt%	15597	

[a]Abbreviations: MDP, 10-methacryloyloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethylmethacrylate; Bis-GMA, bisphenol A diglycidylmethacrylate; TEGDMA, triethyleneglycol dimethacrylate

[b]Procedures: (a) mix equal volumes of primers A and B, (b) apply primer for 30 s, (c) dry with a gentle stream of air, (d) apply adhesive, (e) gently blow air to dry adhesive, (f) light-cure for 20 s

Erupted intact human molars were used after extraction and collected under our protocol No. 725, which was approved by the appropriate institutional review board. Cylindrical cavities with 2/3 enamel and 1/3 dentin margins, 2-mm depth, 3-mm diameter, and C-factor of 3.7 were prepared on the buccal or lingual cervical regions of each molar using a diamond point (# B12, GC Corp., Tokyo, Japan) under copious air-water irrigation.

Each of the 14 cavities was treated with the adhesive Clearfil Liner Bond 2V (Table 1). After this adhesive was cured, the cavities were bulk-filled with Clearfil Photo Bright resin composite or Palfique Estelite resin composite. The resin composites were then polymerized by the conventional light-curing method (600 mW/cm^2 for 60 s). An experimental quartz-tungsten halogen light-curing unit (GC Corp., Tokyo, Japan) that was connected to a slide regulator was used. This light-curing unit had a control system for lamp voltage and adjustable light intensity, which was measured using a curing radiometer (model 100, Demetron Research, Danbury, CT, USA). After light curing, the specimens were stored in the dark for 24 h in water maintained at 37°C. The restorations were finished and polished using wet 600-grit SiC paper. The specimens were thermocycled for 500 cycles between 5°C and 55°C, with a 30 s dwell time.

Marginal seal and cavity wall adaptation

We used the dye penetration test to determine the degree of adaptation to the cavity margins and walls. This dye penetration test was performed by placing a 1.0% acid red propylene glycol solution (Caries Detector, Kuraray Noritake Dental Inc., Tokyo, Japan) at the margin of the restoration for 5 s, followed by rinsing with water and gentle blow-drying. The extent of dye penetration was observed with a stereomicroscope (20× magnification). A photographic record of each specimen was acquired at this stage.

The specimens were then longitudinally cut in half using a diamond saw microtome (Leitz 1600 Saw Microtome, Ernst Leitz, Wetzlar, Germany) under running water, the dye was reapplied to the cavity walls, and images were acquired to observe gaps. In these images, the extent (length) of dye penetration along the cavity margins and walls was measured using a digitizer (KD4300 model, Graphtec Co., Tokyo, Japan). The areas of marginal dye penetration in the enamel were considered to be enamel cracks. The degree of marginal enamel crack formation was then determined as the ratio of the margin stained with the dye divided by the total length of the enamel cavity margin, which was converted to a percentage. The degree of dentin marginal leakage was determined as the extent (length) of dye penetration as a percentage of the total length of the dentin cavity margin. The degree of marginal defect (marginal enamel crack formation and dentin marginal leakage) was determined as the length of dye penetration as a percentage of the total length of the cavity margin. Dye penetration along the cavity walls was calculated as a percentage of the total cavity wall length. This area was referred to as the cavity wall-resin gap. The dye penetration test scores were compared using the Mann-Whitney *U* test.

Finally, the resin-tooth interfaces were observed using an environmental SEM (Quanta 200 FEG, FEI Co., Hillsboro, OR, USA) under conditions of saturated water vapor pressure of 600-650 Pa. We also assessed the relationship between dye penetration test scores and environmental SEM observations.

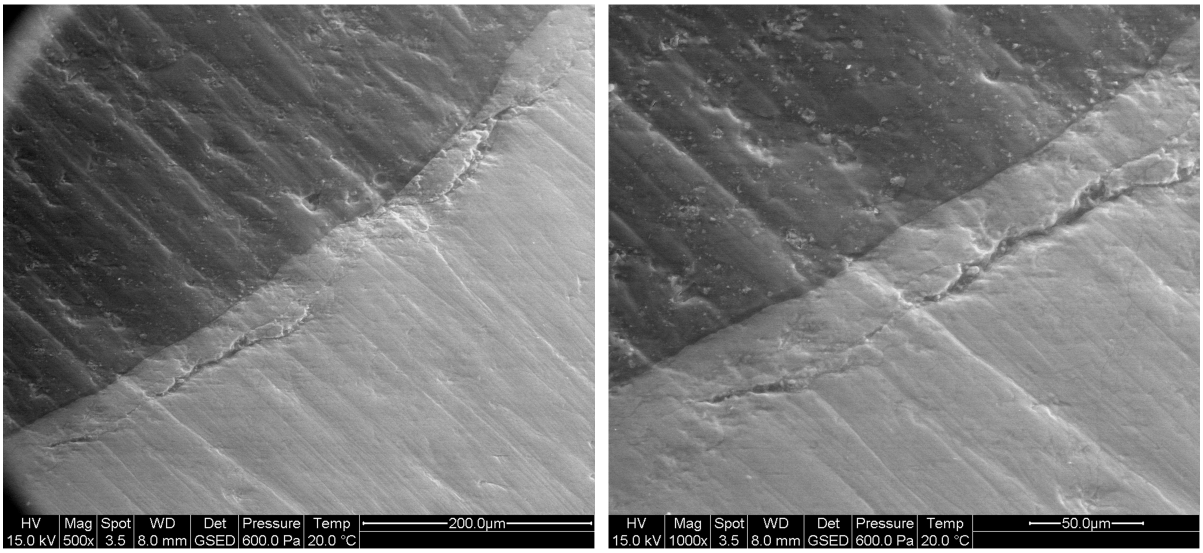
Results

Table 2 shows the results for marginal defect, enamel crack formation, marginal leakage, and cavity wall-resin gap formation. Photographic and environmental SEM observations for the sample specimens are shown in Figs. 1-4. Palfique Estelite showed significantly less dentin marginal leakage compared with Clearfil Photo Bright.

Table 2. Enamel crack formation, dentin marginal leakage, and cavity-wall gap formation [%: mean (SD)]

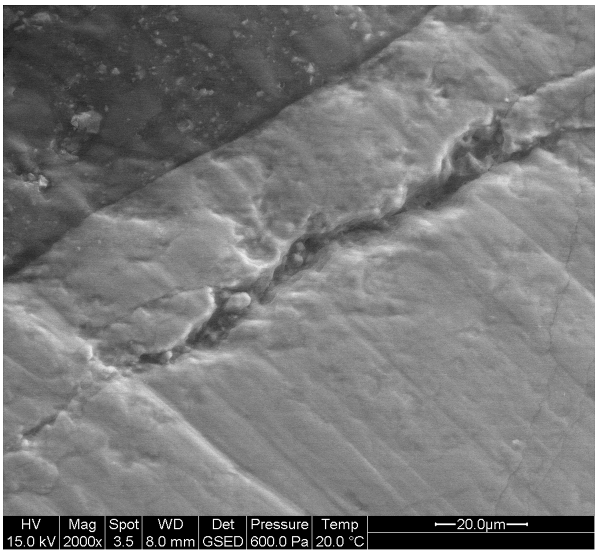
	Clearfil Photo Bright				Palfique Estelite			
Light curing method	Marginal defect	Enamel marginal crack	Dentin marginal leakage	Dentin cavity-wall gap formation	Marginal defect	Enamel marginal crack	Dentin marginal leakage	Dentin cavity-wall gap formation
600 mW/cm^2 60s	36.0 (20.2)	61.9 (41.5)	42.1 (29.3)[a]	40.3 (11.0)	54.6 (10.0)	86.7 (13.5)	4.8 (12.8)[a]	36.2 (13.9)

[a]Intergroup data designed with same superscript small letters for each resin composite are significantly different ($p<0.05$).

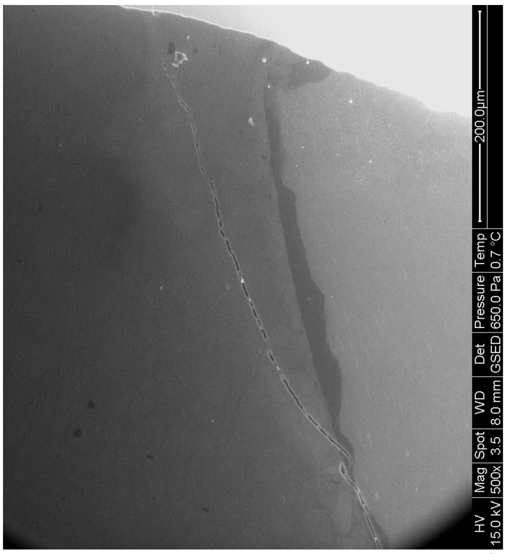


1a 1b

Fig. 1 Environmental SEM observation of enamel cracks on the margins around Palfique Estelite resin composite restorations 1a (500×), 1b (1,000×)



1c



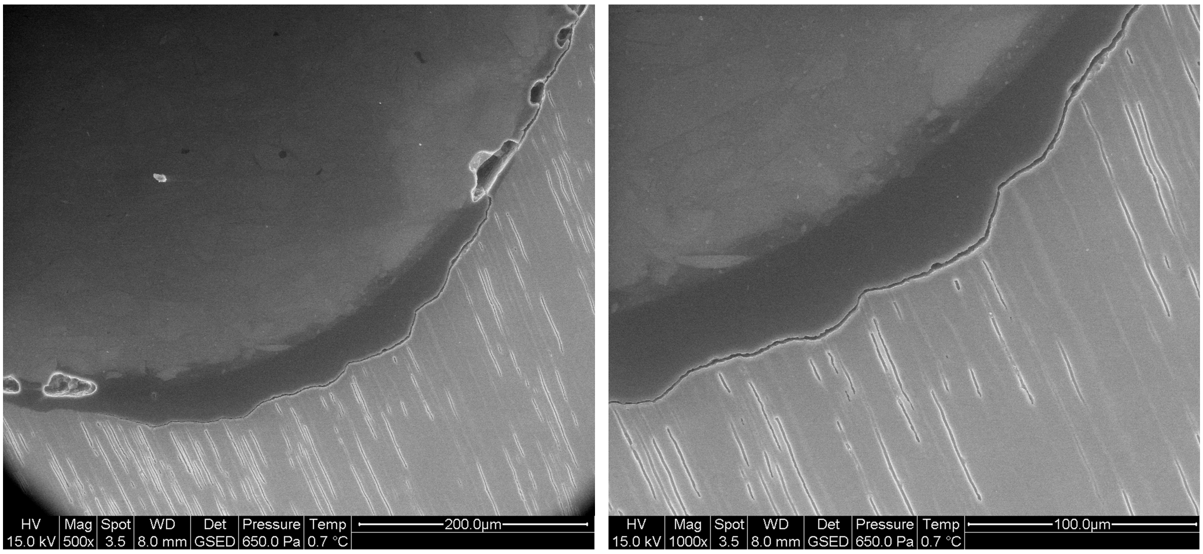
2

Fig. 1c Environmental SEM observation of enamel cracks on the margins around Palfique Estelite resin composite restorations (2,000×)

Fig. 2 Environmental SEM observation of enamel cracks on the cut surface around Palfique Estelite resin composite restorations (500×).

Enamel crack formation was observed in all specimens. In all groups, enamel crack formation appeared as a white margin on a surface that represented cracks in the enamel surrounding the resin composite restorations;

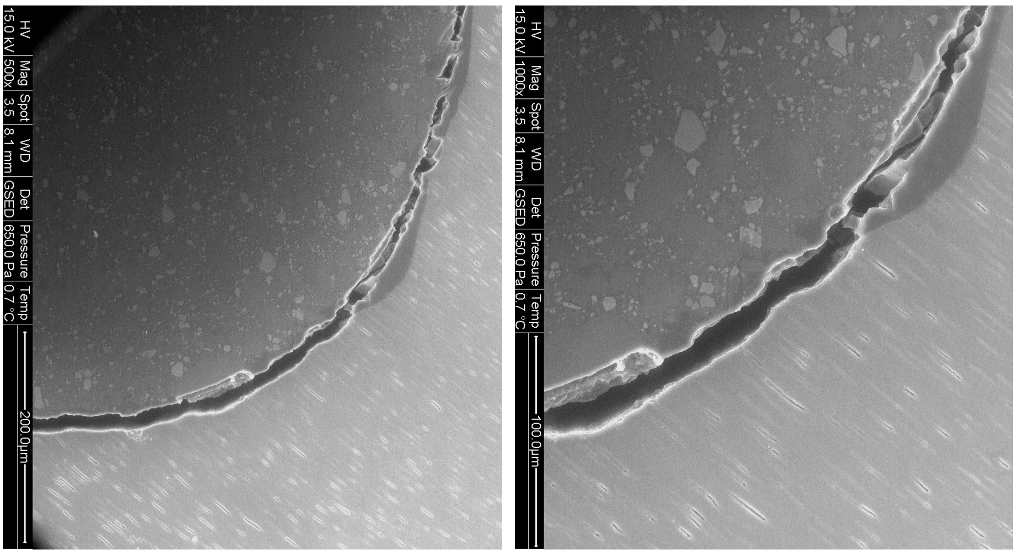
environmental SEM showed that these cracks were located at 25-100 μm from the resin-enamel interface (Figs. 1a, 1b, 1c, 2). Enamel cracks were observed in the enamel surface to the dentin-enamel junction (Fig. 2).



3a 3b

Fig. 3 Environmental SEM observations of cavity wall-resin gap formation in a tooth restored with Palfique Estelite 3a (500×), 3b (1,000×)

Figures 3a and 3b shows an environmental SEM image of a cutting surface obtained after curing Palfique Estelite using the conventional curing method. This shows the environmental SEM image of this gap formation at the line angle between the cavity wall and floor. The gap formation was observed in the dentin-adhesive interface.



4a 4b

Fig. 4 Environmental SEM observations of cavity wall-resin gap formation in a tooth restored with Clearfil Photo Bright 4a (500×), 4b (1,000×)

Figures 4a and 4b show an environmental SEM image of a cutting surface obtained after curing Palfique Estelite using the conventional curing method. This shows environmental SEM images at the line angle between the cavity wall and floor. The gap formation was observed in the resin-bonding interface.

Discussion

In the present study, the conventional light-curing method resulted in poor dentin marginal sealing and dentinal cavity wall adaptation with both Clearfil Photo Bright and Palfique Estelite resin composites.

The curing pattern of light-cured composite resins has several disadvantages that may compromise the ability to achieve an excellent seal along the cavity wall, such as the direction and speed of polymerization shrinkage, depth of curing, and polymerization shrinkage stress. A previous report[19] indicated that at a certain light intensity, the amount of activated starter radicals was optimal to form cross-linked, long-chain molecules. A higher concentration of radicals stopped a reaction earlier than short-chain molecules did, suggesting that curing with a high-intensity light was more likely to result in marginal gaps and poor adaptation of resin composite to the cavity wall.

Polymerization shrinkage that occurs after gelation or curing results in the build-up of large stresses in a resin composite.[20] Conversely, a decreased rate of surface hardness development because of a prolonged gel state and the accompanying absence of dye penetration suggest that this particular protocol results in an increased material flow, which provides for stress relief despite the high elastic modulus and photosensitivity of the resin composite examined.[10]

We also observed enamel crack formation in all of our specimens. Enamel is a highly mineralized tissue, with a modulus of elasticity greater than that of dentin. Enamel prisms are very fragile; the tensile strength of enamel is under 10 MPa when pulled vertical to these enamel prisms[13] The enamel cavosurface reportedly exhibits disruption, even with a decreased power density of 100 mW/cm.[10] Therefore, enamel crack formation was observed in all specimens, when the cavities were prepared with butt-joint margins. Further research is required to determine measures for preventing enamel crack initiation and propagation.

Dye penetration test is useful for both *in vivo* and *in vitro* studies to detect marginal defect and cavity wall gap formation of resin composite restorations when using Caries Detector.

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