Enamel Micro-cracks Produced around Restorations with Flowable Composites

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In this study, enamel micro-cracks produced around flowable composite restorations were observed using a stereomicroscope and a scanning confocal laser microscope (SCLM). The effects of polymerization shrinkage, mobility of composite and polishing period after filling on the incidence of marginal enamel micro-cracks were examined. Enamel micro-cracks were observed on all of the composite restorations when the restoration was polished immediately after filling. Enamel micro-cracks distributed approximately parallel to the cavity margin and located 0.01-0.3 mm from the restored cavity margin. The occurrence of enamel micro-cracks was higher in conventional hybrid composite restorations than in flowable composites, and when polished 15 minutes after filling (as compared to 24 hours after filling).

Key words: Flowable composite, Conventional hybrid composite, Enamel micro-crack

INTRODUCTION

Since the enamel etching technique¹ was first proposed by Buonocore in 1955, many improvements have been made to both adhesive and adhesion technique used in composite resin restorations. Initially, it was tooth surface treatment using 40-60% orthophosphoric acid; presently, phosphoric acid monomer is used in current adhesive systems. Concerning tooth restoration due to caries, the technique has evolved from simple tooth surface decalcification to the current biocompatible treatment technique—which makes the most out of the characteristics of the treated tooth surface²-⁷.

Immediately after finishing and polishing a composite resin restoration, enamel micro-cracks—the so-called white margin—may occur at the enamel cavity margin⁸. Our studies have shown that the leakage occurring through such enamel micro-cracks at the cavity margin and the formation of gaps between the restoration and the cavity wall may cause discoloration around the composite resin restoration, leading to secondary caries and a passing phenomenon of dental pulp irritation or pulpitis³⁰-³². However, despite efforts (such as research and development of new adhesive systems and improvements in existing tooth surface treatment techniques) for more than a decade, flaws such as enamel micro-cracks and micro-leakage at the margin of composite resin restorations are still frequently observed¹¹-¹⁶. Therefore, to achieve an ideal restoration of the cavity without enamel micro-cracks or margin micro-leakage, further improvements are required in terms of both dental material and clinical technique.

The occurrence of enamel micro-cracks is known to be closely related to the polymerization shrinkage of composite resin materials⁹-¹⁰. The polymerization shrinkage of composite resin depends on various factors, such as the content and shape of the fillers⁷-¹⁹. For matrix resin, which is a component of composite resin, Bis-GMA or diluent monomer is used. Polymerization shrinkage is caused by the significant growth of resin molecules when the matrix resin polymerizes. Furthermore, resin polymerization—which occurs extremely rapidly during photoirradiation—causes micro-cracks at the fragile enamel-cavity margin. This is because the polymerization shrinkage stress is focused toward the photoirradiation side, which means that tensile stress is applied to the cavity wall¹⁰-²². There is a tendency that, the higher the resin viscosity, the greater the polymerization shrinkage stress¹⁷,²³.

In recent years, though, resin materials have improved. Flowable composites, which have far lower viscosity than conventional hybrid composites and have been obtained by reducing the filler content and varying the type and composition of monomer, have begun to be used in clinical treatments. It is thought that flowable composites allow less enamel micro-cracks to occur because they flow inside the cavity walls and reach the cavity floor by their own weight due to their excellent flowability, which is in turn due to their low viscosity. As a result, polymerization shrinkage stress is not focused toward the side exposed to light. In this manner, it is hoped that flowable composites will help to curb the occurrence of enamel micro-cracks at the cavity margin after restoration²⁴.

The purpose of this research was to observe the
difference on the occurrence of enamel micro-cracks at the cavity margin when flowable composites and conventional hybrid composites were used. In particular, the factors to consider were the polymerization shrinkage of flowable composites and conventional hybrid composites, mobility of composite resins, and the polishing period after filling.

MATERIALS AND METHODS

Materials

Table 1 shows the composition, manufacturer, and batch number of the composites used in this study. Clearfil Flow FX (FX, Kuraray Medical, Osaka, Japan), Filtek™ Flow (FF, 3M ESPE, MN, USA), PALFIQUE ESTELITE LV Low Flow (PLF, Tokuyama Dental, Tokyo, Japan), and UNIFIL LoFlo Plus (ULP, GC, Tokyo Japan) were used as flowable composites. As controls, two conventional hybrid composites—Clearfil AP-X (APX, Kuraray Medical, Osaka, Japan) and Filtek™ Z250 (Z250, 3M ESPE, MN, USA), a composite of less polymerization shrinkage—were used.

Methods

1. Measurement of coefficient of linear polymerization shrinkage by the mercury bath method

The coefficient of linear polymerization shrinkage of various composite resins was measured using the mercury bath method. As shown in Fig. 1, a mercury bath (box) was fixed on the movable stage of a digital measuring microscope (STM-DH, Olympus, Tokyo, Japan). Using a composite resin syringe with its top cut to a diameter of about 2 mm, a composite resin mud of about 10 mm long was attached to one end of the mercury bath and set afloat on the mercury (Hg). Then, a spherical amalgam alloy

Table 1 Materials, compositions, manufacturers, and batch numbers.

<table>
<thead>
<tr>
<th>Material</th>
<th>Code</th>
<th>Main Composition</th>
<th>Type</th>
<th>Manufacturer</th>
<th>Batch number</th>
</tr>
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<tbody>
<tr>
<td>Clearfil Flow FX</td>
<td>FX</td>
<td>Bis-GMA, TEGDMA UDMA Lanthanide filler Fluoride filler</td>
<td>Flowable type</td>
<td>Kuraray Medical Osaka, Japan</td>
<td>0001AA</td>
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<tr>
<td>Filtek Flow</td>
<td>FF</td>
<td>Bis-GMA, TEGDMA UDMA Titanium Dioxide</td>
<td>Flowable type</td>
<td>3M ESPE, MN, U.S.A.</td>
<td>3BF</td>
</tr>
<tr>
<td>PALFIQUE ESTELITE LV</td>
<td>PLF</td>
<td>Bis-GMA, Bis-MPEPP TEGDMA Titanium Dioxide Silica-Zirconia</td>
<td>Flowable type</td>
<td>Tokuyama Dental Tokyo, Japan</td>
<td>230</td>
</tr>
<tr>
<td>LoFlo Plus</td>
<td>ULP</td>
<td>UDMA Fluoroaluminatesilicate</td>
<td>Flowable type</td>
<td>GC, Tokyo, Japan</td>
<td>310031</td>
</tr>
<tr>
<td>Clearfil AP-X</td>
<td>APX</td>
<td>Bis-GMA, TEGDMA *S-PRG hybrid filler, Radiopacity</td>
<td>Hybrid type</td>
<td>Kuraray Medical Osaka, Japan</td>
<td>009238A</td>
</tr>
<tr>
<td>Filtek Z250</td>
<td>Z250</td>
<td>Bis-GMA, TEGDMA UDMA Nano filler, Silica-Zirconia</td>
<td>Hybrid type</td>
<td>3M ESPE, MN, U.S.A.</td>
<td>2KUJ</td>
</tr>
</tbody>
</table>

*S-PRG: Surface reaction type pre-reacted glass-ionomer
powder (a, b) available on the market was placed on the free end of the cylindrical composite resin specimens. An easily identifiable bright point of this amalgam alloy powder (Spherical D, Shofu, Kyoto, Japan) was set as the reference point of measurement within the visual field of the microscope, thereby measuring the canonical length of the specimen.

The resin was cured using a light-curing unit (XL3000, 3M, MN, USA) according to manufacturer’s instructions. Curing time was 40 seconds and 15 minutes thereafter, the distance was measured again to determine the amount of polymerization shrinkage. All the above measurements were done three times for each composite resin at a room temperature of 21 ± 2 °C, and the mean value was used as the coefficient of linear polymerization shrinkage. The coefficient of linear polymerization shrinkage was computed using the following formula:

Coefficient of linear polymerization shrinkage
\[
= \frac{L_0 - L_1}{L_0} \times 100
\]

where \(L_0\) = Length of specimen before light cure and \(L_1\) = Length of specimen after light cure.

2. Measurement of flowable and conventional hybrid composites in flow

0.2 g of each composite was inserted between two slide glasses, and a 100-g load was added from the top. After 10 minutes, the diameter of each composite material was measured five times, and the average value calculated.

3. Observation of enamel micro-cracks using a stereoscopic microscope

Caries-free, human premolars extracted for orthodontic reasons were stored at 4 °C in isotonic saline not exceeding three months prior to their use in this investigation. Using a round diamond point (# 015, diameter: 1.5 mm; Shofu, Kyoto, Japan), a cylindrical cavity with a diameter of 3 mm and a depth of 1.5-2.0 mm was formed in the center of the buccolingual side of the coronal region. The tooth surface was treated using the test adhesive system appropriate for each resin material. Then, the tooth was filled with composite resin and light-cured for 30 seconds (XL 3000, 3M, Minnesota, USA).

Fifteen minutes after filling, marginal excess was removed with finishing diamond points (# ff C22, Hinatawatda, Tokyo, Japan) and the margin polished with silicon points (# 28, M2 and M3, Shofu, Kyoto, Japan) under a constant water spray as coolant. In addition, another group of specimens were subjected to delayed polishing at seven days after filling. All specimens were then placed in distilled water and stored in an incubator (IC-450, Iuchi Co., Japan) at 37°C. Five specimens for each group were prepared.

After polishing, the specimens of each test group were immersed for 24 hours at 37 in a 0.2% fuchsin solution. Then, enamel micro-cracks at the cavity margin of each restoration were observed under a stereoscopic microscope (SMZ-10, Nikon, Tokyo, Japan). The occurrence rate of enamel micro-cracks was determined using the score method proposed by Han et al.8).

The obtained data were statistically processed by one-way analysis of variance (ANOVA) and multiple comparison test (Tukey HSD) with level of significance set at 5% (n=5).

4. Observation of enamel micro-cracks under a scanning confocal laser microscope

Scanning confocal laser microscope (SCLM, OLS1100, Olympus, Tokyo, Japan) can be adjusted for micro-cracks observation. As such, it was not necessary to preprocess the specimens (i.e., specimens could have a dry surface or be surface-coated).

Specimens after polishing were observed micro-structurally under the SCLM. Scanning confocal laser microscope (SCLM, OLS1100, Olympus, Tokyo, Japan) can be adjusted for micro-cracks observations. As such, it was not necessary to preprocess the specimens (i.e., specimens could have a dry surface or be surface-coated).

Specimens after polishing were observed micro-structurally under the SCLM. Furthermore, the specimens were cut longitudinally so that the longitudinal sections may be observed. Furthermore, the specimens were cut longitudinally so that the longitudinal section may be observed.

RESULTS

Coefficient of linear polymerization shrinkage of composite resins

Fig 2 shows the coefficients of linear polymerization shrinkage of the tested composite resin materials. For the four types of flowable composite tested, no significant differences were observed among their coefficients of linear polymerization shrinkage (p > 0.05). While flowable composites showed slightly higher coefficient of linear in comparison than conventional hybrid composites, no significant differences were observed between these two types of composite resin (p>0.05). On the other hand, Z250 (the so-called composite resin of less polymerization shrinkage) showed the lowest coefficient of linear polymerization shrinkage, and a significant difference was observed between FX and ULP of the flowable composites (p<0.05).

Viscosity of flowable and conventional hybrid composites

Flowable composites had significantly lower viscosity (p<0.05) than conventional hybrid composites (Fig.
Fig. 2 Coefficient of linear polymerization shrinkage on test composite resins.

Fig. 3 Difference in the diameters of flowable and conventional hybrid composites.

Fig. 4 Stereomicroscope pictures of an enamel micro-crack.
A: Polished after filling 15 minutes.
B: Polished after filling 7 days.
Arrow: Enamel micro-crack.
Amongst all tested composite materials, APX ranked the lowest.

**Enamel micro-cracks observation**

Fig. 4 shows the stereoscopic photomicrographs of enamel micro-cracks at the cavity margin in the immediate-polish specimens (parts (A) of Fig. 4) and the delayed-polish specimens (parts (B) of Fig. 4). An observation of the boundary between the enamel cavity and the restoration material revealed that in the immediate-polish group, enamel micro-cracks had occurred partially. Further, an observation of conventional hybrid composite specimens revealed that enamel micro-cracks had occurred continuously on almost the entire circumference of the cavity. As for the delayed-polish specimens, the occurrence of enamel micro-cracks was reduced significantly compared to the immediate-polish ones — for both flowable composites and conventional hybrid composites. In particular, specimen B of FF showed no enamel micro-cracks.

Figs. 5 and 6 show typical examples of enamel micro-crack observed with SCLM. Fig. 5 shows an example of no enamel micro-cracks in the flowable composite FF specimen. Fig. 6, on the other hand, shows the cracks at the enamel-cavity margin in the Z250 specimen. In particular, Fig. 6B shows a fragment of cracked enamel attached to the resin filling material.

For SCLM observation of other specimens, results like those of Figs. 5 and 6 were obtained.

**Occurrence rate of enamel micro-cracks**

Fig. 7 shows the occurrence rate of micro-cracks. In the immediate-polish group, all flowable composites showed occurrence rates of around 30% (i.e., a low value), whereas the conventional hybrid composites Z250 and APX showed occurrence rates of around 80-90%. In other words, a significant difference was observed between the flowable and conventional hybrid composites (p<0.05).

In the delayed-polish group, for both flowable and conventional hybrid composites, the occurrence rate of enamel micro-cracks decreased conspicuously,
and no significant differences were observed between the materials. However, significant differences were observed for all composite resin materials between the immediate-polish group and the delayed-polish group (p<0.05).

DISCUSSION

Since the report published by Jorgensen et al.\(^{25}\), according to which enamel micro-cracks occurred at the cavity margin of composite resin filling materials, numerous investigations have been conducted on this matter. However, no effective ways of preventing the occurrence of micro-cracks have been found. Enamel micro-cracks are the cause of dentin hypersensitivity, pulp symptoms of the restored tooth and secondary caries, as well as esthetic disturbances such as marginal discoloration of the restoration after composite resin restoration. Composite resin contains Bis-GMA as the base resin. However, due to its high viscosity, it is imperative to blend it with a diluent monomer, such as TEGDMA (triethylene-glycol dimethacrylate). However, TEGDMA is of low molecular weight and results in high number of double bonds per unit of weight. This then leads to a high degree of cross-linking, creating a very rigid, stiff composite with a relatively high amount of shrinkage\(^{26}\). This could be an underlying cause to the occurrence of enamel micro-cracks.

One reason why flowable composites are widely used can be ascribed to their low viscosity\(^{27–29}\). The filler content of flowable composites is 56-63% – far lower than the 78-85% of conventional hybrid composites. Accordingly, then, the amount of polymerization shrinkage in conventional hybrid composites is presumed to be great\(^{30–32}\). All the measurement values of linear polymerization shrinkage ratio for the flowable composites tested in this study were greater than those of conventional hybrid composites. However, the occurrence rate of enamel micro-cracks at the cavity margin after restoration was considerably lower in flowable composites than in conventional hybrid composites. In conventional hybrid composites, the polymerization shrinkage stress was generated in the direction of photoirradiation, which had an effect on the occurrence of enamel micro-cracks\(^{33,34}\). Whereas in flowable composites, polymerization shrinkage stress was more likely to be dispersed toward the cavity wall, because even at the moment of polymerization the flow continued toward the cavity floor and the cavity walls under the flowable composite’s own weight. The low occurrence rate of enamel micro-cracks in flowable composites seemed therefore to be related to the low viscosity of these composites (Fig. 8). Moreover, as shown in Fig. 3, flowable composites had a low viscosity compared with conventional hybrid composites and were rich in mobility. Furthermore, as generally shown from the results of this experiment, composites which incurred a low rate of enamel micro-cracks were also rich in mobility. Therefore, it is thought that the composite which is rich in mobility can help to curb the occurrence of enamel micro-cracks.

Moreover, the stress of polymerization shrinkage on flowable composites is low compared to conventional hybrid composites\(^{17,25}\). This is probably be-
cause the stress present at the resin-dentin or resin-enamel interface is buffered by the transformation of the flowable composite.39

While polymerization shrinkage of composite resins is one of the direct causes of enamel micro-cracks, the effect of the strength of shrinkage stress on the cavity wall should also be taken into account, with due consideration to the depth of cavity and the number of surfaces (configuration factor, C-factor).36,37 Dynamically box-form wall surfaces are most vulnerable to stress. In other words, Class I and Class V box-form cavities are most susceptible to the occurrence of enamel micro-cracks around conventional hybrid composite filling materials.39,40

Z250, which is a conventional hybrid composite with low polymerization shrinkage, has managed to remain widely used. It consists of a reduced amount of TEGDMA through the utilization of two base resins: Bis-GMA and UDMA. While the coefficient of linear polymerization shrinkage was somewhat lower than that of APX, a conventional hybrid composite and those of flowable composites, the occurrence rate of enamel micro-cracks around the restoration was not lower than those of flowable composites. The above results seemed to suggest that the high viscosity of conventional hybrid composites, which tended to focus polymerization shrinkage toward the photoirradiation surface, was also likely to have induced enamel micro-cracks.

In the studies heretofore, various measures have been taken against enamel micro-cracking, including an increase in the amount of fillers in composite resins, alteration of the irradiator, adjustment of the irradiation time (soft-start curing, step irradiation)39-42, and improvements to the diluent-monomer blending technique. But even with such measures, the polymerization shrinkage of resin is unavoidable. Investigations into new monomers produced through ring-opening polymerization (with a lower volume-shrinkage ratio during polymerization), cationic polymerization (without oxygen inhibition) are currently being conducted. However, it will take some time to develop into dental materials for clinical use.

Our series of studies have shown that the occurrence rate of enamel micro-cracks in teeth restored with flowable composites is low.40 We suggested that the occurrence of enamel micro-cracks at the cavity margin could perhaps be suppressed by a combined restoration with two materials of different consistencies. In other words, fill the cavity with a conventional hybrid composite after lining the cavity walls and cavity floor with a flowable composite.

Furthermore, this study compared the occurrence rates of enamel micro-cracks between the technique of polishing immediately upon resin filling and that of delayed polishing (i.e., seven days after resin filling). In the immediate-polish group, the occurrence rate of enamel micro-cracks for conventional hybrid composites was greater than that for flowable composites (p<0.05). This was presumed to be directly related to the viscosity of the resin. In the same vein, the adhesion strength of composite resin adhesive agent to enamel also has a close relation. When the teeth were polished one week after filling instead of 15 minutes after filling, the occurrence of micro-cracks and micro-leakage was reduced. Polymerization shrinkage acts as a tensile stress on the marginal enamel. The presence of excessive resin at the cavity margin suppressed the shrinkage of the resin and hence reduced the occurrence of enamel micro-cracks. If the marginal excess resin were to be removed by polishing immediately after filling, the exerted force during polymerization shrinkage, which serves as an internal stress, can cause enamel micro-cracks. Furthermore, if polishing is delayed until one week after filling, hygroscopic expansion occurs in the resin restoration44-46, thereby releasing the internal stress. For these reasons, enamel micro-cracks are less likely to occur when the marginal excess resin is removed one week after filling.

As shown in the pattern diagram of Fig. 8, it is difficult to induce micro-leakages and enamel micro-cracks at the cavity margin in low-viscosity, easy-flow flowable composites. This is because the polymerization shrinkage which occurs at the cavity wall or cavity floor due to the flow of resin is compensated for and the polymerization shrinkage stress is dispersed. This is possible because polymerization stress is not focused toward the surface of photoirradiation, unlike the shrinkage stress in conventional hybrid composites.

Meanwhile, regarding the delayed-polish specimens, both conventional hybrid composites and flowable composites showed low value in marginal micro-cracks. In other words, there were significant differences between them (p>0.05). This is presumed to be the result of the easing of polymerization shrinkage stress, due to the expansion subsequent to water absorption by the resin material. Therefore, it is highly recommended to avoid any polishing on the day of resin filling, as this seems to be one means of suppressing enamel micro-cracks.

The SCLM images in Fig. 5 show a flowable composite (FF) adhering to enamel with no micro-cracks, whereas the SCLM images in Fig. 6 clearly show micro-cracks in Z250 specimen. Fig. 6B shows an SCLM image of a fragment of cracked enamel at the cavity margin attached to the composite resin. Thus, enamel micro-cracks occurred even when adverse condition, such as the drying of the specimen, had been eliminated.

The results of this study suggested that, in order to suppress enamel micro-cracks as far as possible while restoring caries teeth, it is important to improve the clinical techniques and to use various...
dental materials in a coordinated and harmonious manner, as well as to select materials with due consideration to the polymerization shrinkage characteristics of the composite resin\(^{41-49}\).

CONCLUSION

While flowable composites showed relatively high polymerization shrinkage ratio, the occurrence rate of enamel micro-cracks in restored teeth was lower in flowable composites than in conventional hybrid composites. The occurrence of enamel micro-cracks is usually closely related to the polymerization shrinkage of composite resins, but the effects of the directional character and stress focus of polymerization shrinkage on the cavity wall are dependent on the viscosity of the composite resin. Delayed polishing after filling the cavity based on the observations in this study, is an effective means of suppressing the occurrence of enamel micro-cracks.

REFERENCES