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Study on Polymerization Contraction Stress of Bulk-fill Resin Composites

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Abstract

Purpose: While incremental filling is recommended for deep cavities when a light-cured composite resin（CR）is used, because of issues such as polymerization contraction stress and irradiation depth, bulk-fill CRs, which can be filled in large cavities at once, have been developed and used in clinical application. This study examined the effects of the polymerization contraction stress of bulk-fill CRs in high C-factor cavities by conducting visual evaluation using polycrystallized glass blocks, shear bond strength tests on bovine dentin, and measurement of the residual volume after curing.

Materials and methods: Bulk Base Hard（BH）and Beautifil Bulk Flow（BF）were used as bulk-fill CRs, and Gracefil Flow（GF）as a conventional CR. As bonding systems, Clearfil Mega Bond2（MB）and Clearfil Universal Bond Quick ER（ER）were used. Bioram-M was used as the polycrystallized glass block. The groups filled with each CR after bonding treatment with MB were designated as the MBH Group, MBF Group, and MGF Group, and the groups filled with each CR after bonding treatment with ER as the EBH Group, EBF Group, and EGF Group. A cylindrical cavity of 4.5 mm diameter and 4.0 mm depth in Bioram-M was formed, the cavity was repaired, and the state of gap and crack generation was classified and scored. Furthermore, a flat dentin surface on a bovine front tooth was prepared, repaired, and then the tensile-shearing strength was measured immediately after bonding.

A rubber ring jig with 4.5 mm diameter and 4.0 mm height was prepared and each CR was filled in a darkroom to prepare cylindrical samples. The specimen was soaked in acetone immediately after curing, and the unpolymerized parts of the CR were removed. The residual volume of each specimen was measured after removing the unpolymerized CR.

Results: As a result of visual evaluation, a significant difference in the state of gap and crack generation was observed in the MBH and MBF Groups compared to the MGF Group, and in the EBH and EBF Groups compared to the EGF Group（$p<0.05$）. The MGF and EGF Groups showed gaps at the cavity floor. In addition, the bond strength of the MBF Group was significantly higher than those of the MBH and MGF Groups（$p<0.05$）. The bond strengths of the EBF Group and EGF Group were significantly higher than that of the EBH Group（$p<0.05$）. Although the residual volume of BH and BF were significantly larger than those of GF, there was no significant difference between BH and BF（$p<0.05$）.

Conclusion: The results indicated that care is needed during the filling operation of bulk filling with bulk-fill CRs, as the problems caused by polymerization contraction stress are not completely eliminated in high C-factor cavities, even though it is effective for the bonding of deep cavities.

Key words: bulk-fill composite resin, polycrystallized glass block, polymerization contraction stress, C-factor

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Introduction

Today, light-cured composite resins (CRs) are used for repairs in various dental treatments, as the range of their clinical application has been expanded through improvements in adhesiveness to dentin and mechanical properties[1,2]. However, problems such as coloring on the edges, separation or fracture of repair material, and contraction gap, which are caused by the polymerization contraction of CRs, have emerged[3,4]. The contraction stress caused by polymerization contraction not only causes cracks and gaps to form on the adhesive interface and deteriorates the bond strength, but also causes some effects on the dentin[5,6]. It has also been reported that the effects of polymerization contraction increase in cavities with a high value of Configuration-factor (C-factor), as proposed by Feilzer, et al.[7], namely cavities in which the bonding area is larger than the unbonded area of the repair material, and that the bond strength deteriorates as the depth of the cavity is larger even if the C-factor is low[8]. To overcome the limitation in polymerization depth of light-cured CRs and minimize the polymerization contraction stress, incremental filling is recommended for large and high C-factor cavities with depths exceeding 2.0 mm[9–12]. Bulk-fill CRs, which can be filled at once in large cavities with depths exceeding 2.0 mm, have been developed and used in clinical application in recent years, since a reduction in chairside treatment period and mitigation of the burden for the patient can be expected by simplifying the treatment method[13–15]. However, there have been only a few reported studies on the effects of the polymerization contraction stress caused by restoration using bulk-fill CRs to deep and high C-factor cavities.

In this study, we conducted a visual evaluation using polycrystallized glass blocks which can bond with CRs, shear bond strength tests on bovine dentin, and measurement of the residual volume of CR block immediately after curing through dissolution in acetone to examine visually the effects of the polymerization contraction stress on bulk-fill CRs in deep and high C-factor cavities.

Materials and Methods

1. Materials for experiments

Table 1 shows the light-cured CRs and bonding systems used in this study. For light-cured CRs, Bulk Base Hard (BH; Sun Medical Co., Ltd., Moriyama, Japan) and Beautifil Bulk Flow (BF; Shofu Inc., Kyoto, Japan) were used as bulk-fill CRs, and Gracefil Flow (GF; GC Corp., Tokyo, Japan) as a conventional CR. As bonding systems, Clearfil Mega Bond2 (MB; Kuraray Noritake Dental Inc., Tokyo, Japan) and Clearfil Universal Bond Quick ER (ER; Kuraray Noritake Dental Inc) were used. Bioram-M (Nippon Electric Glass Co., Ltd., Otsu, Japan) was used as the calcium phosphate-based polycrystallized glass blocks, and PenCure (J. Morita Mfg. Corp., Kyoto, Japan) was used as the LED light irradiation device. For classification of the specimens after bonding treatment in each experiment, the ones that were filled with each CR after bonding treatment with MB were designated as the MBH Group, MBF Group and MGF Group, and the ones that were filled with each CR after bonding treatment with ER as the EBH Group, EBF Group and EGF Group.

2. Experiment methods

1) Visual evaluation using Bioram-M

Visual evaluation was conducted using Bioram-M according to the method of Kawamura[16]. Bioram-M was polished from #60 to #600 with waterproof abrasive paper to prepare a flat surface. A cylindrical cavity of 4.5 mm diameter and 4.0 mm depth (C-value=5.0) was formed on the flat surface using diamond point #211 for FG (Shofu Inc) and diamond point #18a Class C for low-speed HP (Shofu Inc) under irrigation (Fig. 1). After conducting ultrasonic cleaning for 5 minutes, bonding was conducted using bonding system MB or ER according to the manufacturer's instructions. Each CR was filled in bulk to irradiate light with PenCure for 20 seconds to cure the CR, then the specimens were soaked in water at 37°C for 24 hours after the bonding procedure. The specimen was cut using a low-speed diamond saw (Model 650, South Bay Technology Inc., California, USA) so that it went through the center of the cavity, and the cut surface was polished from #600 to #2000 with waterproof abrasive paper. Then the state of gap and crack generation was observed with a laser scanning confocal microscope VK-X100 Series

Table 1 Materials used

	Materials	Code	Composition	Manufacturer	Lot No.
Composite resin	Bulk Base Hard (Universal)	BH	Methacrylic acid esters (Bis-MPEPP, etc.), Acrylic acid esters (urethane acrylate), Barium silica glass, Strontium silica glass, Aromatic amines, etc.	Sun Medical	VG11
	Beautifil Bulk Flow (Universal)	BF	Glass powder, Bis-GMA, UDMA, Bis-MPEPP, TEGDMA, Reaction initiators, Colorants, etc.	Shohu	022041
	Gracefil Flo (A2)	GF	Barium glass, Bis-MEPP	GC	2002171
Bonding agent	Two-step self-etch adhesive Clearfil Mega Bond2	MB	Primer：10-MDP, HEMA, Hydrophilic aliphatic dimethacrylates, DL-CQ, Water, Accelerators, Dyes Adhesive：10-MDP, HEMA, Bis-GMA, Hydrophilic aliphatic dimethacrylates, DL-CQ, Colloidal silica, New initiators, Accelerators	Kuraray Noritake Dental	000098
	One-step self-etch adhesive Cleafil Universal Bond Quick ER	ER	10-MDP, Bis-GMA, HEMA, Hydrophilic amide monomer, Colloidal silica, Ethanol, DL-CQ, Accelerators, Water, Sodium fluoride	Kuraray Noritake Dental	BCO234

(CLSM; KEYENCE Corp., Osaka, Japan). The state of gap and crack generation was classified and scored according to the method of Kawamura[16] (Fig. 2). The number of specimens was set to 10 in each group, and statistical processing using the Kruskal-Wallis test and the Steel-Dwass test ($p<0.05$) was conducted.

2) Shear bond strength tests

As test teeth, bovine front teeth that had been frozen for storage after extraction were used by defrosting them immediately before the experiment. After preparing the flat dentin surface with a modeling trimmer, the bonding surface was prepared by polishing it up to #600 with waterproof abrasive paper. A piece of double-sided tape with a hole of 3.0 mm inner diameter was placed on the bonding surface, then a black nylon tube of 2.5 mm inner diameter and 4.0 mm height was fixed to specify the bonding area. The bonding procedure was conducted according to the manufacturer's instructions using bonding system MB or ER. Each CR was filled in bulk and cured by irradiating light with PenCure for 20 seconds. Immediately after the bonding procedure, the shear bond strength was measured using a universal testing machine AUTOGRAPH AGS-J 5 kN (SHIMADZU Co., Kyoto, Japan) at CHS=1.0 mm/min. The number of specimens was set to 10 in each group. Statistical processing was conducted using one-way analysis of variance and Tukey's test ($p<0.05$).

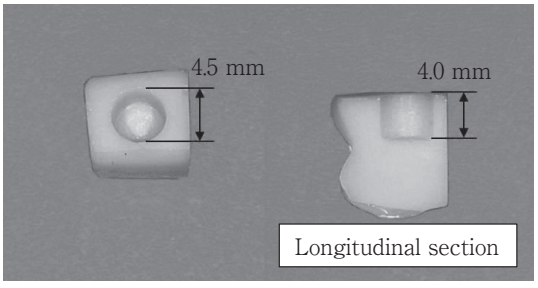

Fig. 1 Bioram-M
A cavity of 4.5 mm diameter and 4.0 mm depth was formed in Bioram-M, which is the polycrystallized glass block. It can bond with CR via bonding systems.

This experiment was conducted after receiving approval from the Animal Care and Use Committee, Osaka Dental University (Approval No. 20-04009).

3) Measurement of cured volume

A rubber ring jig with 4.5 mm diameter and 4.0 mm height was prepared and each CR was filled in bulk in a darkroom. The cylindrical sample was prepared by irradiating light with PenCure for 20 seconds with the specimen pressure-welded with a slide glass. The specimen was soaked in acetone immediately after curing, and subjected to ultrasonic cleaning for 60 seconds to remove the unpolymerized parts of the CR. The residual volume of each specimen was measured after

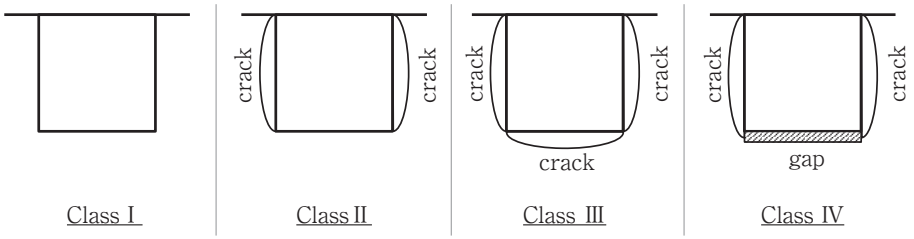

Fig. 2 Classification of gap and crack formation

Class Ⅰ：Specimens with no cracks in the proximity of the cavity or gaps at the cavity floor. Class Ⅱ：Specimens with some cracks on the side walls but no gaps or cracks at the cavity floor. Class Ⅲ：Specimens with cracks on the side walls and cavity floor. Class Ⅳ：Specimens with cracks on the side walls and gaps at the cavity floor. No other states were observed.

removing the unpolymerized CR with CLSM. The number of specimens was set to 10 in each group. Statistical processing was conducted using one-way analysis of variance and Tukey's test ($p<0.05$).

Results

1. Visual evaluation using Bioram-M

As shown in Figure 2, the results of gap and crack generation state were classified as described below.

Class I: Specimens with no cracks in the proximity of the cavity or gaps at the cavity floor.

Class II: Specimens with some cracks on the side walls but no gaps or cracks at the cavity floor.

Class III: Specimens with cracks on the side walls and cavity floor.

Class IV: Specimens with cracks on the side walls and gaps at the cavity floor.

No other states were observed.

Table 2 shows the score results when using MB as the bonding system, and Figure 3 the typical CLSM observation image on the longitudinal section of each. Table 3 shows the results using ER, and Figure 4 the typical CLSM observation image on the longitudinal section of each.

As a result of the Kruskal-Wallis test, there were significant differences in the results of the gap and crack generation state in each group using MB or ER as the bonding system ($p<0.05$).

Many Class III specimens were observed in the MBH and MBF Groups, and many Class IV specimens in the MGF Group. As a result of the Steel-Dwass test, the MBH and MBG Groups had significantly more Class III specimens than the MGF Group regarding the state of gap and crack generation ($p<0.05$). Cracks at the cavity floor were observed in the MBH and MBF Groups, and gaps at the cavity floor in the MGF Group. Furthermore, many Class III specimens were observed in the EBH and EBF Groups and Class IV specimens in the EGF Group. As a result of Steel-Dwass test, the EBH and EBF Groups had significantly more than the EGF Group regarding the state of gap and crack generation in a similar fashion to the specimens using MB ($p<0.05$).

Table 2 Distribution of crack and gap formation with MB

	Class Ⅰ	Class Ⅱ	Class Ⅲ	Class Ⅳ
MBH	0	0	10	0
MBF	0	0	9	1
MGF	0	0	0	10

］: Significant difference ($p<0.05$)

While CLSM observation images in the MBH and EBH Groups showed cracks within Bioram-M on the side wall and at the cavity floor, they showed no gaps between Bioram-M and BH. Similarly, the images showed no gap between Bioram-M and BF in the MBF and EBF Groups, even though they showed cracks within Bioram-M on the side wall and at the cavity floor. In the MGF and EGF Groups, the images showed cracks within Bioram-M on the side wall and gaps between Bioram-M and GF at the cavity floor.

2. Shear bond strength tests

Figure 5 shows the shear bond strength of the groups that used MB, and Table 4 the distribution of failure modes on the fracture surface. Figure 6 shows the shear bond strength of the groups that used ER, and Table 5 the distribution of failure modes on the

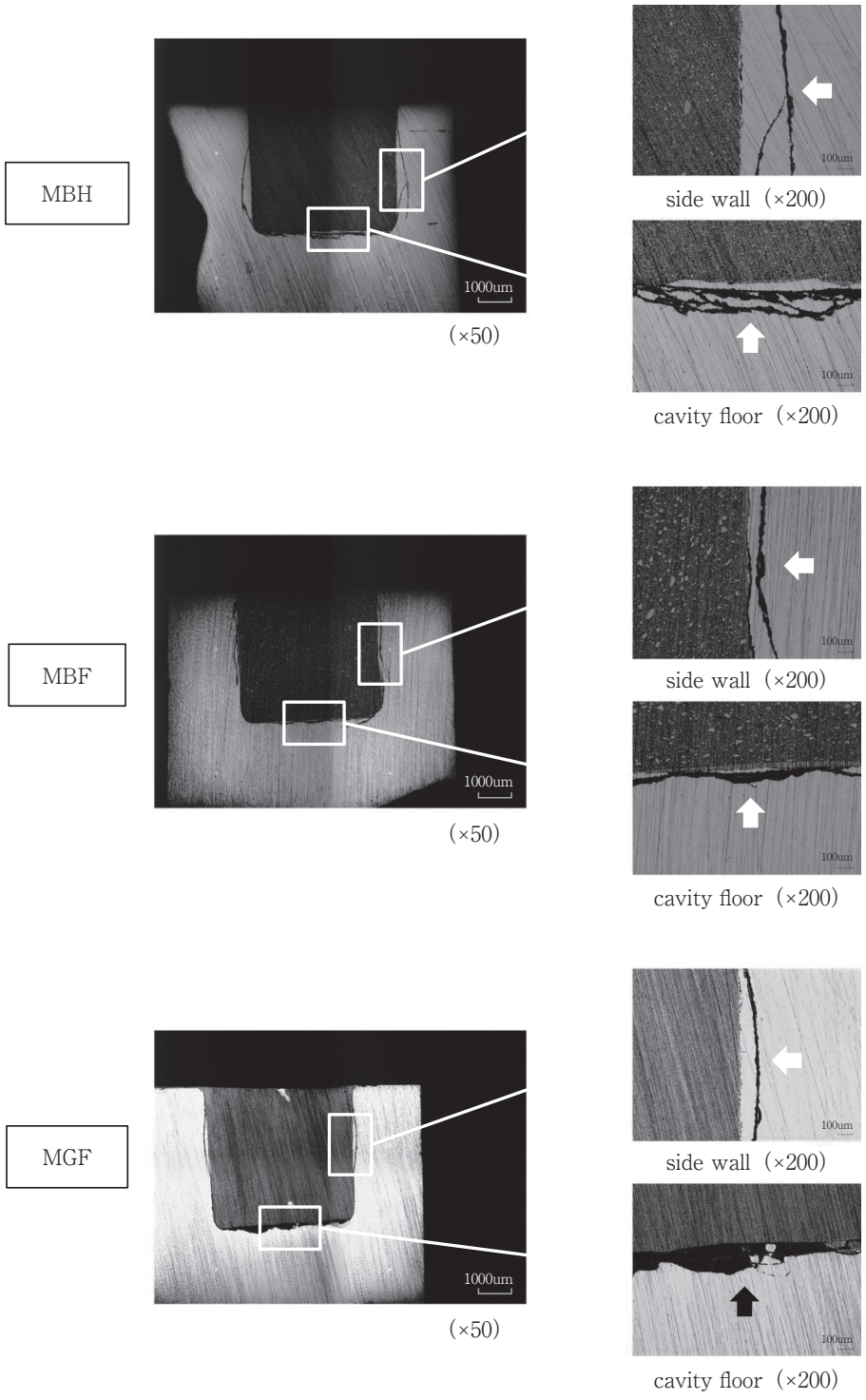

Fig. 3 CLMS images of longitudinal cross-sectional surface on the whole, side wall and cavity floor with MB
Overall image (×50), side wall part (×200) and cavity floor part (×200) of CLSM observation image.
MBH and MBF Groups : Cracks (white arrows) within Bioram-M were observed on the side wall part and the cavity floor part, and no gap between Bioram-M and BH. MGF Group : Cracks (white arrows) within Bioram-M were observed on the side wall part, and gaps (black arrow) between Bioram-M and GF were observed at the cavity floor part.

Table 3 Distribution of crack and gap formation with ER

	Class I	Class II	Class III	Class IV
EBH	0	0	10	0
EBF	0	1	8	1
EGF	0	0	3	7

]: Significant difference ($p<0.05$)

fracture surface.

The shear bond strengths of each group that used MB were 6.4 (±0.9) MPa in the MBH Group, 10.0 (±0.9) MPa in the MBF Group, and 6.9 (±2.6) MPa in the MGF Group. While the bond strength of the MBF Group was significantly higher than those of the MBH and MGF Groups ($p<0.05$), there was no significant difference between the MBH Group and MGF Group ($p>0.05$). Although there were many mixed failures in the MBH and MGF Groups, the numbers of specimens with mixed failure and dentin cohesion failure were nearly equivalent in the MBF Group. The shear bond strengths of each group that used ER were 5.5 (±0.9) MPa in the EBH Group, 7.5 (±0.9) MPa in the EBF Group, and 7.0 (±1.5) MPa in the EGF Group. While the bond strengths of the EBF Group and EGF Group were significantly higher than that of the EBH Group ($p<0.05$), there was no significant difference between the EBF Group and EGF Group ($p>0.05$). While there were many bonding cohesion failures in the EBH and EBF Groups, there were many mixed failures in the EGF Group.

3. Cured volume

Figure 7 shows the results of residual volume measurement after dissolving the unpolymerized layer of CR in acetone solvent. The volume before dissolution was 63.6 mm^3. The volume after dissolution was BH 62.2 (±0.8) mm^3, BF 62.5 (±0.9) mm^3, and GF 60.4 (±1.7) mm^3, respectively. Although the residual volume of BH and BF were significantly larger than those of GF ($p<0.05$), there was no significant difference between BH and BF ($p>0.05$). In addition, clouding and cracks that seemed to have been caused by dissolution in acetone were observed by the naked eye on the cavity floor on the other side of light irradiation in GF.

Discussion

The polycrystallized glass blocks used as the adherends in this experiment were pieces of amorphous glass in which crystals such as apatite and diopside had been deposited by thermal processing, and had properties similar to natural enamel[17,18]. This material has been reported to show adhesive properties with CR by mechanical interlocking force when acid treatment and phosphate ester-based bonding agents are used[17,19]. In addition, it is considered appropriate as a material to examine the effects of polymerization contraction, as there are few individual differences among adherends, it can bond stably with CR, and it is possible to visually confirm the strain based on the cracks formed in the adherend by polymerization contraction due to its high brittleness. Our preliminary experiment was also conducted with each of the bonding systems used in this experiment. Each bonding system in the experiment was used after confirming that it showed sufficient adhesion to polycrystallized glass blocks of around 15 MPa, and that no cracks were generated by the cutting procedure.

GF, which is a conventional CR, had many Class IV specimens in which cracks were generated in side walls and gaps at the cavity floor regardless of the bonding system in visual evaluation using Bioram-M. In addition, the shear bond strength of GF was equivalent to that of BH when MB was used, and it was significantly lower than that of MBF. When ER was used, the shear bond strength was significantly higher than that of BH but equivalent to that of BF. The cured volume of GF after soaking in acetone was significantly smaller than those of bulk-fill CRs. Since it has been widely reported that the cured depth of conventional CRs is approximately 2.0 mm[20,21], it is indicated that the light did not reach and that curing was insufficient in the depths of deep cavities exceeding 2.0 mm. Furthermore, the formation of gaps at the cavity floor suggests that it was pulled in the direction of light irradiation due to the effect of polymerization contraction occurring from the direction of light irradiation before it bonded with the bonding system[16]. Therefore, the results indicate that restoration with the conventional CR in deep cavities exceeding 2.0 mm was affected by the cured depth and the polymerization contraction.

Meanwhile, bulk-fill CRs enable bulk filling even in deep cavities exceeding 2.0 mm by alleviating the restriction on curing depth of light-cured CRs through changes in filler properties, filler content, and constitu-

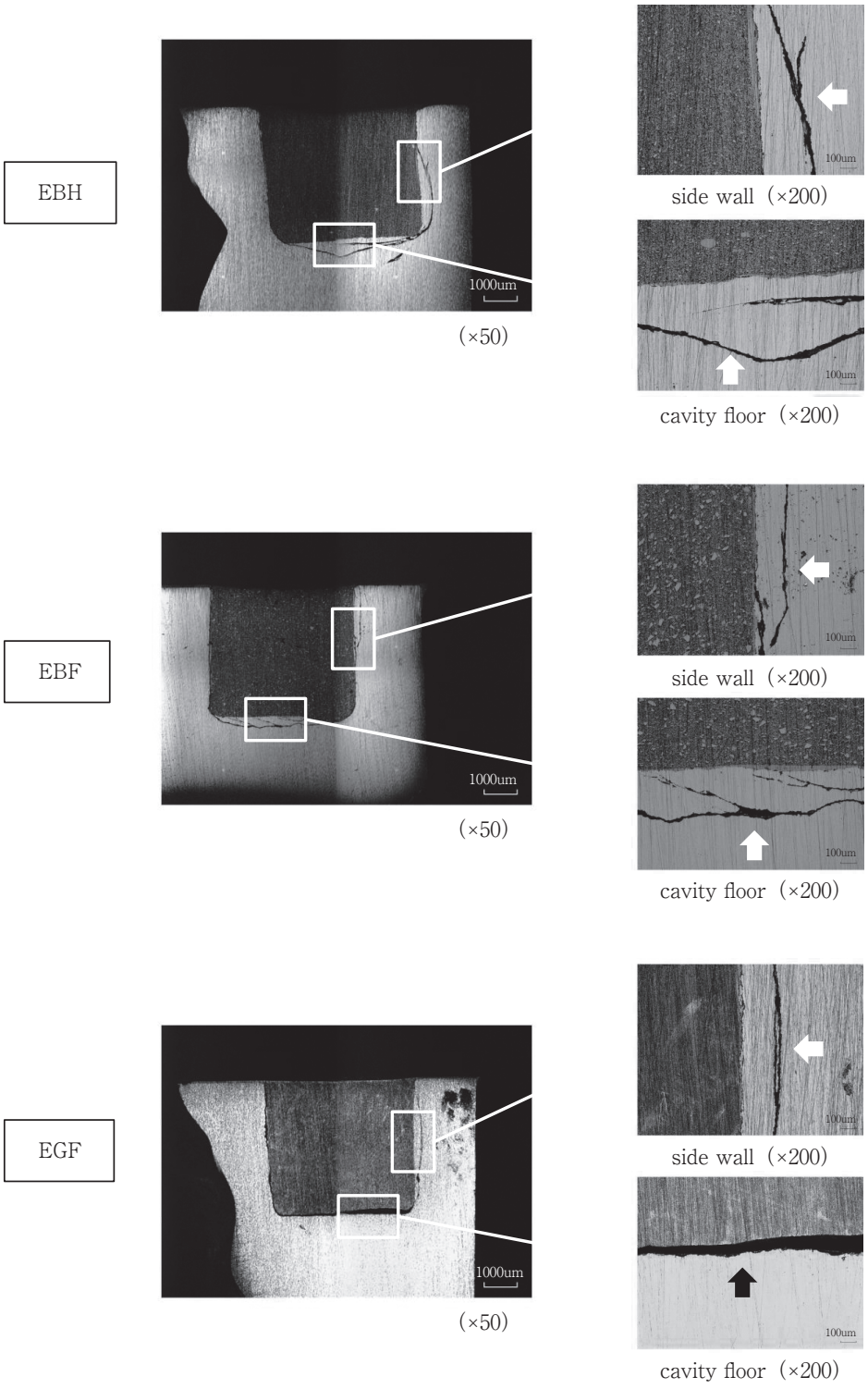

Fig. 4 CLMS images of longitudinal cross-sectional surface on the whole, side wall and cavity floor with ER
Overall image (×50), side wall part (×200) and cavity floor part (×200) of CLSM observation image.
EBH and EBF Groups : Cracks (white arrows) within Bioram-M were observed on the side wall part and the cavity floor part, and no gap between Bioram-M and BH. EGF Group : Cracks (white arrows) within Bioram-M were observed on the side wall part, and gaps (black arrow) between Bioram-M and GF were observed at the cavity floor part.

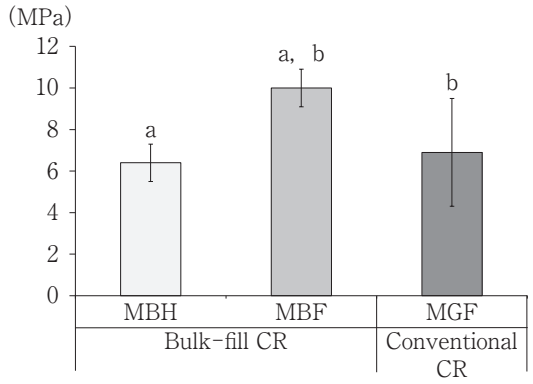

Fig. 5 Shear bond strengths of each resin composite with MB to dentin
Same letters indicate significant differences ($p<0.05$).

Table 4 Failure mode of MB

	Bonding cohesion failure	Interface fracture	Mixed failure	Dentin cohesion fracture
MBH	0	0	10	0
MBF	0	0	5	5
MGF	1	1	7	1

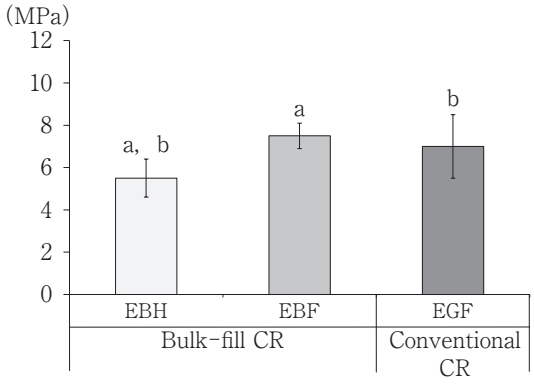

Fig. 6 Shear bond strengths of each resin composite with ER to dentin
Same letters indicate significant differences ($p<0.05$).

ent monomer components[22,23].

Class III, in which cracks were generated on the side walls and cavity floor, was observed in all BH specimens regardless of the bonding system in visual evaluation using Bioram-M. The shear bond strength of BH was significantly lower than that of BF. It was equivalent to that of GF when MB was used, and significantly lower than that of GF when ER was used. The results suggest that the shear bond strength of BH was low because this study did not use the accessory primer containing hydrophilic polymerization initiator, whose action helps BH to cure from the contact interface[24]. However, the cured volume of BH after soaking in acetone was equivalent to that of BF, and significantly larger than that of GF. BH has characteristics such as low shrinkage factor thanks to the low-shrinking monomer contained[25,26], long flow period of CR on a free surface, and slow progress of the polymerization reaction[27]. It is indicated that the time difference since the start of light irradiation until the generation of contraction stress becomes longer and leads to the reduction in contraction stress if the flow period is long, as stress is not generated while there is flow on the free surface of CR during curing[28].

Similarly to BH, most of the specimens of BF, which is also a bulk-fill CR, were Class III regardless of the bonding system. The shear bond strength of BF was

Table 5 Failure mode of ER

	Bonding cohesion failure	Interface fracture	Mixed failure	Dentin cohesion fracture
EBH	10	0	0	0
EBF	10	0	0	0
EGF	3	0	7	0

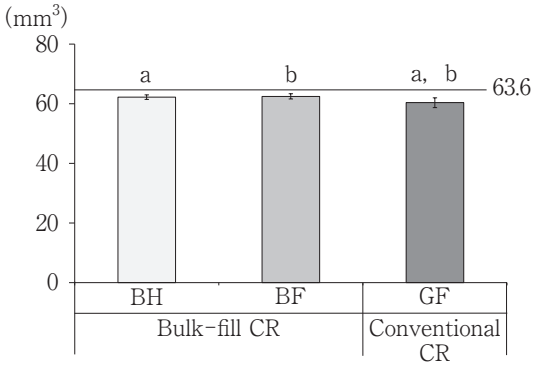

Fig. 7 Volume of each resin composite after dissolving the non-polymerization layer dissolution with acetone
Same letters indicate significant differences ($p<0.05$).
The volume before dissolution was 63.6 mm^3.

significantly higher than that of BH. In addition, it was equivalent to that of GF when ER was used, even though it was significantly higher than that of GF when MB was used. The cured volume after soaking in acetone was also significantly larger than that of GF. BF contains a S-PRG filler and it is indicated that the effect of light irradiation reaches into deep parts thanks to its high light permeability and light diffusion property[29,30]. It is also indicated that the progress of the polymerization reaction is fast, based on a report that polymerization contraction stress increased rapidly after light irradiation[27]. The results suggest that BF and BH, which are bulk-fill CRs, polymerize sufficiently even in deep parts and bond with the bonding materials in deep cavities with depths exceeding 2.0 mm.

Based on the above results, bulk-fill CRs seem to be more effective than the conventional CRs in improving the contraction gaps at the cavity floor which occur in the case of bulk filling of deep cavities. However, these results also indicated that careful filling operations are necessary in a similar fashion to the conventional CRs in order to prevent problems such as white margin, as the effects of polymerization contraction cannot be completely eliminated in large and high C-factor cavities with depths exceeding 2.0 mm.

Conclusion

This study examined the effects of the polymerization contraction stress of bulk-fill CRs in high C-factor and deep cavities by conducting visual evaluation by using polycrystallized glass blocks, shear bond strength tests to dentin, and measurement of the residual volume of CR block immediately after curing through dissolution in acetone. The following conclusions were obtained:

1. Bulk filling using bulk-fill CRs bonded to cavity floor and generated no contraction gaps in deep cavities of depth 4.0 mm.

2. While bulk filling using bulk-fill CRs is effective for repairing deep cavities, careful filling operations are necessary as the problems caused by polymerization contraction stress cannot be completely eliminated in high C-factor cavities.

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The authors declare no conflicts of interest associated with this manuscript.

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バルクフィルコンポジットレジンの重合収縮応力に関する研究

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抄録

目的：光硬化型コンポジットレジン（以下，CR）修復では重合収縮応力や照射深度の問題から，深い窩洞には積層充塡が推奨されているが，大型窩洞に対して一括充塡できるbulk-fill CRが開発され臨床応用されている．今回，多結晶化ガラスブロックを用いた視覚的評価，せん断接着試験ならびに硬化後の残存体積の測定を行い，C-factorの大きな窩洞におけるbulk-fill CRの重合収縮応力の影響について検討を行った．

材料と方法：bulk-fill CRとしてBulk Base Hard（BH）とBeautifil Bulk Flow（BF）を，従来型CRとしてGracefil Flow（GF）を，接着システムとしてClearfil Mega Bond2（MB）とCleafil Universal Bond Quick ER（ER）を用いた．多結晶化ガラスブロックとして，Bioram-Mを使用した．MBで接着処理後，各CRを充塡した群をMBH群，MBF群，MGF群とし，ERで接着処理後，各CRを充塡した群をEBH群，EBF群，EGF群とした．Bioram-Mに直径4.5 mm深さ4.0 mmの円柱窩洞を形成し，修復後，ギャップおよびクラックの発生状態を分類し，スコアリングを行った．また，ウシ歯前歯に象牙質平坦面を作製し，修復後，接着直後のせん断引張強さを測定した．

直径4.5 mm，高さ4.0 mmのゴムリング治具を作製し，暗室にて各CRを充塡し，円柱試料を作製した．硬化後すぐアセトンに浸漬し，CRの未重合部分の除去を行った．各試料の残存体積を測定した．

結果：視覚的評価の結果，MBH・MBF群はMGF群と，EBH・EBF群はEGF群と比べて，ギャップやクラックの発生状態に有意な差が認められた（$p<0.05$）．MGF・EGF群では窩底部にギャップが認められた．またMBF群の接着強さはMBH・MGF群より有意に高かった（$p<0.05$）．EBF群とEGF群の接着強さはEBH群と比べて有意に高かった（$p<0.05$）．BHとBFの残存体積はGFの残存体積よりも有意に大きかった（$p<0.05$）が，BHとBFの間に有意差は認められなかった．

結論：bulk-fill CRを用いた一括充塡は深い窩洞における接着には有効であるが，C-factorの大きな窩洞においては重合収縮応力による不快事項が完全には解消せず，充塡操作に留意する必要があることが示唆された．

キーワード：バルクフィルコンポジットレジン，多結晶化ガラスブロック，重合収縮応力，C-factor

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