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Effects of fiber reinforcement on adaptation and bond strength of a bulk-fill composite in deep preparations

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ABSTRACT

Objective. This study investigated the effect of plasma-treated leno weaved ultra-high-molecular-weight polyethylene fiber placement on gap formation and microtensile bond strength (MTBS) of a bulk-fill composite in deep cavity.

Methods. Resin composite molds (3 mm width, 4 mm depth) were treated with Clearfil SE Bond 2 and restored with 3 techniques : (1) Surefil SDR flow (SDR) placed in bulk (BLK), (2) SDR placed in two unequal increments (INC) and (3) SDR placed after an increment of SDR placed with wetted polyethylene fiber (Ribbond Ultra) at the cavity floor (FRC). As a control, the cavities were bulk-filled with SDR and no bonding agent (n = 12). All the specimens were subjected to real-time and 3D imaging by SS-OCT (1330 nm) to calculate the total volume of gap formed (mm^3) at the cavity floor and between the composite increments. For MTBS, the occlusal cavities of the similar dimensions ($3 \times 3 \times 4$ mm^3) were prepared on extracted molars with similar composite placement techniques (BLK, INC and FRC). After 24 h 37 °C water storage, the specimens were sectioned using a diamond saw to create 0.7×0.7 mm^2 beams for MTBS, and subjected to bond testing at a crosshead speed of 1 mm/min. Data for both tests was analyzed by one-way ANOVA and multiple-comparisons with Bonferroni correction ($\alpha = 0.05$).

Results. The gap volumes were different among the groups ($p < 0.05$). The largest cavity floor gaps (mm^3) were observed in the control group (2.00 ± 0.08); followed by BLK (0.74 ± 0.20) and INC (0.02 ± 0.01). In FRC, the cavity floor was gap-free in all specimens but some separation was observed between the two increments. MTBS values (MPa) were 13.8 ± 7.6, 31.7 ± 12.5 and 28.3 ± 8.5 for BLK, INC and FRC groups. There was no significant difference between FRC and INC and both were different from BLK ($p < 0.05$).

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Significance. Gap formation of the bulk-fill composite at cavity floor was significantly reduced with the placement of a fiber-reinforced increment at the base of the deep preparation. The fiber-reinforced increment acts as a shrinkage stress breaker and protects the bonded interface at deep dentin.

1. Introduction

Resin composite has witnessed a rapid growth in utilization for posterior teeth to correspond to aesthetic and functional demand [1]. Adhesive systems are used to provide efficient bond between resin composites and the tooth structure, which is critical for the success and longevity of aesthetic restorations [2]. Many factors such as the bonding substrate, bonding area, preparation geometry, technique of composite application, light curing efficiency and occlusal force equilibration contribute to gaining clinical success with direct posterior resin restorations [3]. The shrinkage of methacrylate-based composite resins as direct result of polymerization is a widely researched phenomenon in literature [4–7]. The shrinkage inevitably occurs as the resin monomers in the paste form move closer to each other and link covalently to form the polymer.

If the internal stresses arising from shrinkage forces exceed the resisting forces at the cavity internal surfaces, it would lead to the loss of sealing at the interface between the tooth and composite [8]. The polymerization shrinkage stress in composite resins may result in marginal disintegration, cuspal deflection, enamel crack formation, reduced bond strength, compromised mechanical properties, and interfacial gaps between composite and tooth structure, all of which are factors accounting for the success or failure of a composite restoration. The stress could be strong enough to immediately initiate a crack within the restorative material itself [9].

Modifying restorative techniques may reduce the stress of polymerization shrinkage. It is not clear which restorative technique, if any, is the most appropriate one to eliminate shrinkage stress. Incremental application of composite instead of a bulk technique has been proposed to reduce stress of shrinkage in cavities [10]. However, the incremental technique in restoring deep preparations is time-consuming and relatively technique sensitive [10], and some reports have even proposed that they have little effect on reducing the total shrinkage stress [11]. Recently, bulk-fill composites, which can be inserted as a single increment layer 4–5 mm in thickness have been proposed as an alternative to simplify the restorative procedure and decrease polymerization shrinkage stress [12–14]. Provided that the quality of the resulting restoration is comparable with or surpasses that of the incremental technique, the bulk-fill composite would seem to be a preferred technique for restoring deep cavities [15].

Nevertheless, bonding to cavity floor dentin may be challenging in large deep composite restorations, even using state-of-the-art bulk-fill composites [16–18]. Using optical coherence tomography (OCT) imaging technique, Hayashi et al. reported that all the tested light-cured bulk-fill composites lost adaptation to a 4-mm deep cavity floor due to polymerization shrinkage [16].

Since shrinkage is an intrinsic resin property, reducing resin volume by adding non-monomer components such as organic or non-organic fillers has been considered as an effective way to reduce the magnitude of shrinkage [9]. Fiber-reinforced composite (FRC) is a material that carries such an effect on polymerization shrinkage, while enhancing physical properties of the composite and potentially acting as a crack stopping mechanism [19–22]. The mechanical properties and reinforcing capacity of FRC used in dentistry depend on the fiber type, fiber orientation relative to the load, fiber position in the restoration, fiber volume and impregnation of the fiber to the resin matrix [20].

The use of fiber-reinforced composites have been broadly evaluated in various studies [20–22]. Chairside incorporation of ultra-high molecular-weight polyethylene fiber (UHMWPE) into resin composite has received a renewed attention for the direct restoration of structurally compromised teeth that require the use of a large amount of composite [21]; however, the combination of UHMWPE fiber and bulk-fill composite resin in deep cavities has not been evaluated. The current study investigated whether an increment of composite placed with plasma-treated leno-weaved UHMWPE fiber (Ribbond Ultra, Ribbond Inc, Seattle, WA) at the base of a deep cavity affected the debonding of composite from the cavity floor, total gap formation in the composite and the microtensile bond strength (MTBS) of composite to deep cavity dentin. The null hypotheses were that the gap formation at the cavity floor and the bond strength were not affected by the placement technique.

2. Materials and methods

2.1. *Experimental design*

This study consisted of two major experiments: adaptation of the composite to 4-mm deep molds using OCT imaging and MTBS of the composite to deep dentin in a restored preparation in human teeth. The bulk-fill composite (SDR; SDR flow, Dentsply Sirona, DE, USA) was used in combination with the UHMWPE fiber.

2.2. *Adaption to cavity floor*

In order to standardize the cavity geometry and ensure adequate OCT signal intensity to observe composite adaptation at the cavity floor, a standard composite mold was created using flowable composite (Estelite Flow Quick, Tokuyama Dental, Tokyo, Japan) [16]. The internal surfaces of the molds (3

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mm width and length, 4 mm depth,) were sand-blasted with 50 μm alumina particles (MicroEtcher II; Danville Materials, Carlsbad, CA, USA) at 0.4 MPa for 15 s, followed by ultrasonic cleaning for 10 min (Model US 102; SND, Nagano, Japan), phosphoric acid cleaning (K-etchant gel; Kuraray Noritake Dental, Tokyo, Japan), silanization (Clearfil Ceramic Primer Plus; Kuraray Noritake Dental, Tokyo, Japan) and bond application (CSE; Clearfil SE Bond 2 Bonding Agent; Kuraray Noritake Dental, Tokyo, Japan). Light curing was proceeded for 20 s using a halogen light curing unit with 600 mW/cm^2 light irradiance (Optilux 501, Kerr, Orange, CA, USA) according to the manufacturer instruction. The specimens were allocated into 3 groups according to placement techniques (n = 12). In group 1 (BLK), SDR was placed with a syringe tip in bulk and cured for 20 s. In group 2 (INC), bulk-fill composite was applied in two unequal increments (approx. 0.3 mm and 3.7 mm in thickness) and each increment was light cured for 20 s. In group 3 (FRC), SDR was placed after an increment of composite reinforced with UHMWPE fiber as follows; a thin layer of SDR bulk-fill composite (similar to INC) was applied on the cavity floor with a syringe tip without curing. A 3 mm × 3 mm piece of fiber was cut and soaked in CSE bonding agent; the excess of the bonding was removed by gently tapping a dry micro brush on fiber, then the fiber was placed over the bulk-fill composite in the cavity and gently pushed through the uncured composite to create a FRC increment. The increment was manipulated with a non-sticking plastic hand instrument so that the layer of fiber was laminated as closely as possible against the cavity floor. This combination was cured for 20 s, creating an approximately 0.3 mm thick FRC increment. Finally, the cavity was bulk filled with SDR and cured for 20 s. As for a control group, the cavities were bulk-filled with SDR without any primer or bonding agent treatment of the mold.

2.3. OCT imaging and analysis

The swept-source OCT system (Prototype 2, Panasonic Healthcare Co., Ltd., Ehime, Japan) was used to monitor the internal integrity of the specimen during the light curing of all groups; BLK, INC, FRC and control group in 2D real-time videos as well as 3D imaging of the cavity after polymerization. The prototype system has been presented with details [23]. A center wavelength at 1330 nm with a 30-kHz sweep rate was used in this system. The transversal resolution is 20 μm and axial resolution is 12 μm in air. It also provides a photographic image of the specimen. 3D scan was obtained after light curing at 256 × 256 × 1024 pixels over an XYZ volume of 5 × 5×7 mm^3. The experimental set up for deep cavity imaging has been reported in detail [16]. For analysis of the volume of gaps formed, the OCT 3D data were imported to Amira software (version 5.5.0, FEI Visualization Science Group). Using the labelling function of the 3D image analysis software and based on the increased reflectivity at the gap boundaries [16], the gap volume was calculated in two stages in mm^3; first for the cavity floor only and then for the whole restoration, which included both the floor and the composite increments. The data were analyzed by one-way ANOVA for the two types of gaps; the cavity floor and the whole restoration.

2.4. Cross-sectional microscopy

Furthermore, confocal laser scanning microscopy (CLSM, 1LM21H/W, Lasertec Co., Yokohama, Japan) was used for cross-sectional microscopy. The specimens of each group were sectioned and mirror-polished up to 0.25 μm with diamond pastes in circular motion under cooling water to physically reach the axial cross-section imaged in 2D real-time videos. The cavity wall and floor on each cross section were observed at 1250 magnification using CLSM. The representative specimens were further sputter gold-coated and observed under scanning electron microscopy (SEM; JSM-6010PLUS/LA, JEOL, Tokyo, Japan).

2.5. Bond strength to cavity floor dentin specimens

For the MTBS test, the occlusal cavities were prepared on the human teeth. Twenty-four intact human mandibular molars were collected after extraction for periodontal, orthodontic or surgical reasons. Before proceeding the study, teeth were examined to ensure they were intact, non-restored and non-carious. From the time of extraction, teeth were stored in water at 4 °C for less than two months. The teeth were mounted in epoxy resin (Total Boat, Bristol, RI) and the cusps were sectioned with a low-speed diamond saw (Buehler, Lake Bluff, IL) under abundant cooling water to form an occlusal flat surface. To ensure reaching a 4-mm deep preparation without exposure of the pulp chamber or too thin remaining dentin thickness, a 1-mm thick layer of hybrid resin composite (AP-X, Kuraray Noritake Dental, Tokyo, Japan) was placed on the occlusal flat surface and cured for 20 s with CSE according to the manufacturer's instructions.

An occlusal cavity (3 × 3×4 mm^3) was prepared in each tooth using a long flat-end cylindrical diamond bur (Shofu, Kyoto, Japan) under cooling water to obtain a flat cavity floor. The cavities were treated using CSE according to the manufacturer instructions and restored with 3 placement techniques similar to the cavity adaptation study to obtain 3 experimental groups: BLK, INC and FRC.

2.6. Microtensile bond test

After 24 h of storage in 37 °C water, teeth were sectioned using a low speed diamond saw under abundant cooling water to create 0.7 × 0.7 mm^2 beams. The marginal beams and beams with remaining dentin thickness of less than 2 mm were excluded from this study. Four central beam with no defects were selected for the MTBS test at a crosshead speed of 1 mm/min (Bisco Microtensile Tester, Schaumburg, IL). A bond strength value of zero was recorded for central beams with pre-test failure. For statistical analysis purposes, each tooth was considered as a statistical unit and therefore, all the values obtained from each tooth were averaged (n = 8). One-way ANOVA with post-hoc Tukey HSD was used to compare MTBS at a significance level of alpha = 0.05. After MTBS test, the dentin side of the beams was collected and the mode of failure was observed by SEM.

3. Results

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Table 1 – OCT Gap volume at cavity floor and whole restoration (Mean ± SD, mm³).

Location	BLK	INC	FRC	Control
Cavity floor	0.74 ± 0.20 [A]	0.02 ± 0.01 [B]	0.0 ± 0.0*	2.00 ± 0.08 [C]
Whole restoration	0.72 ± 0.18 [D]	0.39 ± 0.06 [E]	0.27 ± 0.06 [F]	2.00 ± 0.08 [G]

Different letters indicate statistically significant difference in each row with one-way ANOVA and multiple-comparisons with Bonferroni correction ($p < 0.05$). BLK: bulk filled with SDR; INC: two increments of SDR; FRC: ribbond fiber reinforced increment followed by SDR.
* Mean and standard deviation value of 0 were excluded from statistical analysis.

The volumetric gap values are reported in Table 1, and representative OCT images are presented in Fig. 1. One-way ANOVA showed that the placement technique significantly affected the volume of gap formed after polymerization ($p < 0.05$). When considering the cavity floor only, the largest gap was seen in the control (unbonded) group. All other groups showed some gap formation at the cavity floor except for FRC, where the cavity floor was gap-free in all specimens. There was a significant difference between INC, BLK and control ($p < 0.05$). When the whole restoration void including the gap between increments was considered, there was no significant difference between FRC and INC ($p > 0.05$). No further voids were observed within the bulk-filled BLK and control groups. Real-time videos are presented online (BLK and FRC), with key screen shots in the video still. CLSM images (Fig. 2) confirmed that in BLK, the separation occurred at the bonded interface while in FRC, it happened between the two increments. CLSM confirmed close adaptation of SDR to the fiber within the FRC increment, which appeared to be around 0.3 mm in thickness.

Regarding the bond strength, MTBS values were 13.8 ± 7.6, 31.7 ± 12.5 and 28.3 ± 8.5 (mean ± SD; MPa) for BLK, INC and FRC groups (Fig. 3). While there was no significant difference between FRC and INC, both were different from BLK ($p < 0.05$). No pre-test failures were observed in FRC group. SEM micrographs showed that the separation under load in BLK and INC was predominantly at the dentin-adhesive interface (adhesive or mix failure mode), while in FRC it always involved the fiber-reinforced increment (Fig. 4), and dentin interface was not exposed in any MTBS beams post-processed. Fig. 3 presents predominant failure modes after MTBS test.

4. Discussion

OCT has enabled unique experimental setups to study gap formation at the deep cavity floor with this type of composite. The imaging depth of OCT through composite is limited depending on the optical properties [23], and imaging at 4-mm cavity depth would require a modified imaging technique;

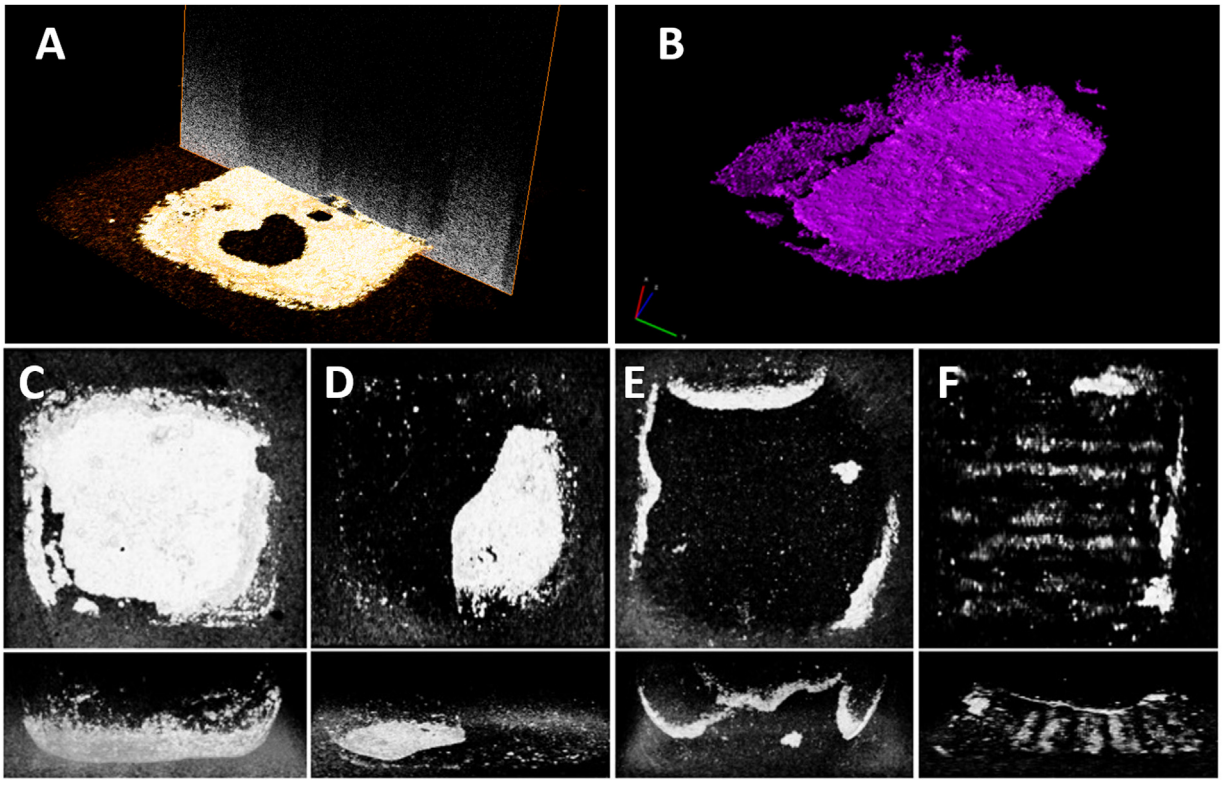

Fig. 1 – OCT images obtained from cavity bottom; A: the 3D images were processed to isolate the gap B: the volume of gap at the bottom was calculated based on the reflectivity from the gap boundaries. C–F: enface OCT images of composite separation from cavity bottom. The bright white areas indicate interfacial gaps. The lower pane displays the side view; C: control; D: BLK; E: INC; F: FRC. The woven pattern of fiber can be observed in F.

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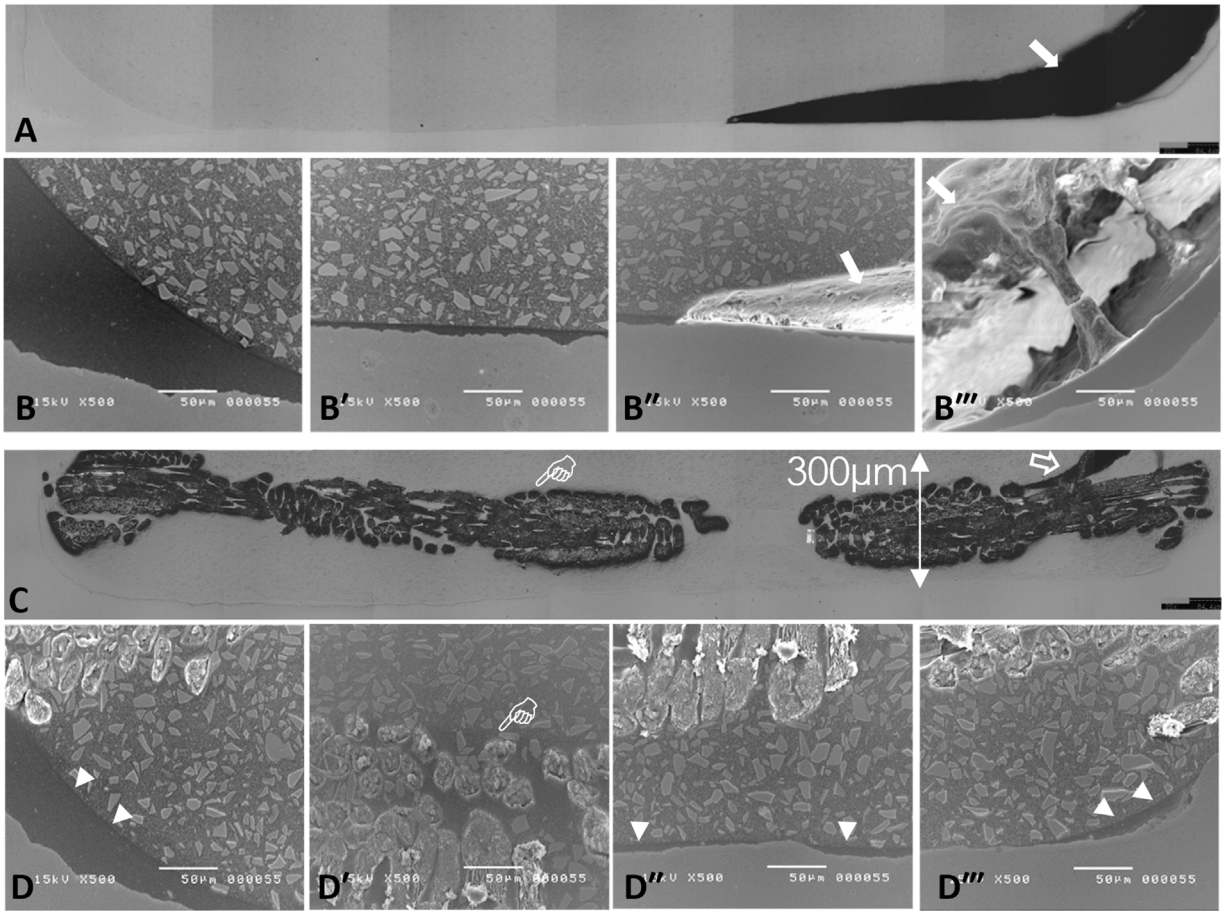

Fig. 2 – Cross-sectional CLSM and SEM images of composite adaptation at cavity bottom. These images correspond to OCT images presented in the video. In BLK and FRC groups. A: CLSM image of the cavity floor in BLK shows debonding of SDR at the line angle. B, B′, B″ and B‴: SEM images of the cross section in A, it appears that polymerization shrinkage has pulled away the composite from the bonding layer (bold white arrow). C: CLSM image of the cavity floor in FRC shows that fiber-reinforced increment reached a thickness of approximately 0.3 mm. A gap can be observed between the bulk placed SDR and FRC increment in C (blank arrow). D, D′, D″ and D‴: SEM images of the cavity floor cross section presented in C, good adaptation of SDR with fiber increment at all interfaces can be observed (arrowheads). The resin impregnated fiber is well integrated with composite (finger pointer).

such as physically grinding the superficial composite to reduce depth after curing [18] or using a relatively translucent composite mold and image the floor through the bottom of the mold [16]. The use of a composite mold should be seen as the limitation of this experimental setup, since bonding of resin to dentin and stress development in a tooth cavity would be different and less consistent that those in a composite mold. Polymerization-induced stresses will mostly be present even with low-shrinkage bulk-fill composites. The results of the current study confirmed that the stress developed in the 4-mm bulk fill flowable was strong enough to debond the composite from the cavity floor in the OCT experiment and result in poor bond strength to the deep cavity dentin in the MTBS experiment. It has been shown that large shrinkage vectors develop at the deeper parts of by a bulk-filled light-cured resin composite in bonded cavities result in debonding [24]. This shrinkage gradient through depth of placement can be explained by the light attenuation associated with the absorption and scatter within the material. In addition to the material and polymerization factors, the vectors can depend on cavity configuration, shape and compliance [16].

In this study, the bond strength in both FRC and INC groups was significantly higher than BLK. These observations were corroborated by previous studies suggesting that despite improvements made in the current light-cured bulk-fill composites in terms of curing depth and lower polymerization shrinkage stress, bulk filling a deep cavity may cause bond detachment at the bottom of the restoration [17]. Using SDR, it was previously reported that cavities 2 mm in depth produced significantly better adaptation than those with 4 mm depth [25]. It was also shown that SDR placed in <2-mm thick increments produced higher MTBS to the gingival wall of deep MOD preparations [26]. Therefore, placement of a lining increment prior to bulk-filling with SDR is a clinical recommendation to be advised for deep preparations.

Meanwhile, OCT showed no debonding from the cavity floor in any of the specimens in FRC group, and none of the MTBS beams separated at the dentin interface under the load.

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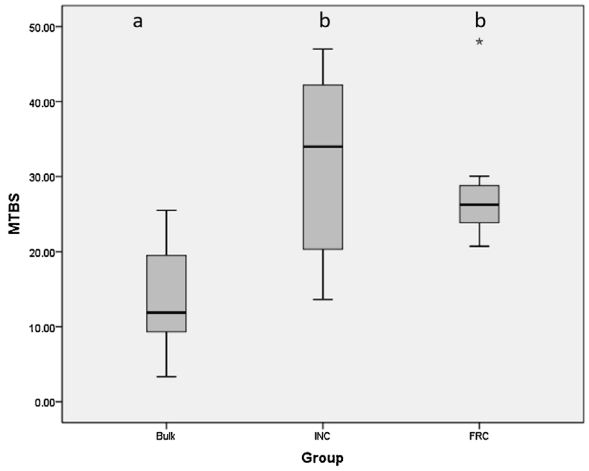

Fig. 3 – MTBS values to deep dentin for BLK, INC and FRC groups. Data was averaged per tooth as a biological unit. Different letters indicate statistically significant difference with one-way ANOVA and multiple-comparisons with Bonferroni correction ($p < 0.05$). The horizontal bars within the box plots represent median, box presents from the first quartile to the third quartile and the whiskers extend from each quartile to the minimum or maximum. Outlier data point is presented by (*).

The UHMWPE fibers may have a modifying effect on the development of interfacial stress along the boundary of resin and tooth substrate [27]. The addition of FRC increment in the current study can resemble the creation of a fail-safe design for restoration of teeth; in a fail-safe system, the polymerization shrinkage stress will not result in debonding of composite from dentin or deformation of the tooth. Ideally, the shrinkage energy would be completely absorbed in the fiber system. While placing a thin layer of the bulk-fill flowable composite in INC group as a liner created better results than bulk-filling alone in BLK group, there are key differences between the FRC and INC groups which highlight the function of FRC layer. These findings suggest that the UHMWPE fiber composite layer possibly mitigated the shrinkage stresses that would have otherwise resulted in debonding or deformation at the pulpal floor. In line with these assumptions, minimal stress values within UHMWPE FRC was reported using finite element (FE) method for stress analysis [28]. A recent FE analysis study demonstrated the advantage of the two-layer restorative technique in 4-mm deep cavities over bulk fill technique, when the first layer was assumed to be a non-shrinking glass ionomer [29].

A fractography study on MTBS has shown that lining the cavity with a flowable composite increased the bond strength; however, did not affect crack formation with dentin under the MTBS mechanical stress compared with the group that did not use a lining [30]. It is assumed that the FRC increment would decrease crack formation by debonding stress. However, due to the location of FRC beam separation, the presented values of MTBS are not representative of the actual MTBS of composite bonded with CSE to dentin, but rather indicate a cohesive strength variable within the FRC. According to OCT observation, the inevitable shrinkage stress of the bulk-fill composite resulted in internal separations in FRC group, with total void values that were comparable to those of the INC technique. It is

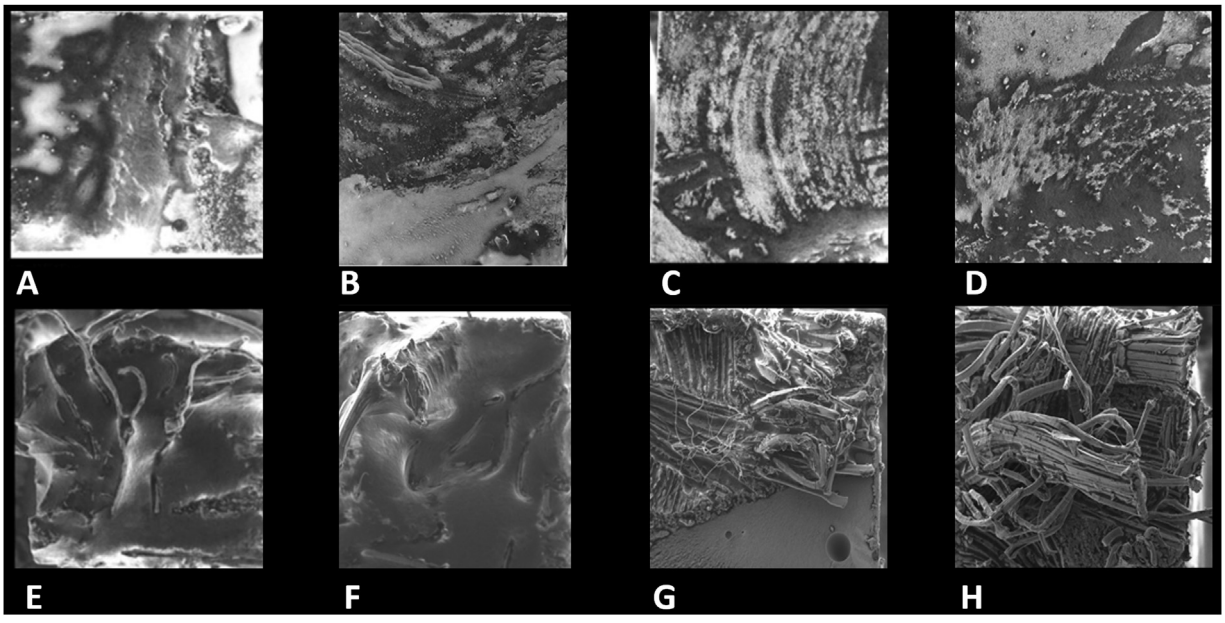

Fig. 4 – SEM micrograph of dentin side of microtensile beam after test at ×150 magnification. A, B: BLK; C, D: INC; E–F: FRC. A: adhesive failure between the composite and bonding layer in BLK; B: dentin tubule orifices are exposed with adhesive failure between the bonding layer and dentin in BLK; C: dentin tubule orifices are exposed with adhesive failure between the bonding layer and dentin in INC; D: a mix with debonding at the cavity floor from dentin and composite in INC, dentin is partly exposed. E–H: The resin-embedded fiber is exposed in FRC at various levels. In G, composite underneath the fiber in visible indication separation beneath the fiber. In H, the fiber network seems to be not completely wetted with the resin, in contrast to E and F. In none of FRC beams, separation occurred at dentin or bonding layer interface.

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assumed that an internal separation involving the FRC increment such that as observed in the current study would safely occur in a region that will not result in leakage, hypersensitivity or pulp complications. Nevertheless, if the fiber completely debonds from the resin prior to function, it may not reach its limit of energy absorption under the load; therefore, its effectiveness would be reduced.

The OCT real-time video for BLK demonstrates that the gap at the floor starts about 5 s after the irradiation and propagation continued afterwards. A similar finding was found for the bulk-fill composite in a previous report [16]. The gap analysis in this study was performed immediately after light-curing. Real-time imaging showed that the majority of shrinkage gaps from dentin occurred during light-curing of the composite, and once these gaps formed, they continued to propagate after the light-curing period [31]. The residual stress from the postgel shrinkage could result in cuspal deformation and propagation of cracks in the tooth structure [31]. Moreover, thermal and mechanical stresses would result in crack propagation through the contemporary composite resins [32]. Therefore, study on defect development in dental restoration should consider the time factor. Several static-load studies have confirmed that a UHMWPE FRC layer improves the fracture strength of direct composite restorations [33–35]. Future studies should consider the fatigue loading and long-term performance of these restorations.

The strength of bond between fiber and resin in a FRC system is vitally important. The bond has to be weak enough to allow the crack stopping and energy absorption mechanism to work, and create a fail-safe system to protect the tooth interface from debonding stresses. Yet, the fiber-resin bond must be strong enough to carry the load from one fiber to another and keep the integrity of the restoration. Moreover, the impregnation of reinforcing fiber with resin is critical for the strength of composite, because it promotes the integration of the fiber with the polymer matrix. Insufficient impregnation such as voids can cause oxygen to inhibit radical polymerization of the acrylic resin inside the composite, resulting in higher residual monomer content in the fiber composite and reduced FRC strength [36]. Interestingly, the nominal MTBS values in FRC were close to that of the INC group, but the failure in INC group was predominantly involving the dentin interface. From the current MTBS result, the FRC increment in the current study seems to meet the fiber-resin strength criteria discussed above. The SEM images confirmed that the fiber impregnation and placement steps are critical steps that contribute to the effectiveness of the FRC restorative technique. Previously, it was suggested that the dense network structures and small-diameter fibers of an UHMWPE fiber (Ribbond THM) made impregnation of the fibers with resin difficult [37]. The current UHMWPE fiber is less thick than its predecessor (120 μm vs. 180 μm) and therefore appears to be more impregnable and easier to adapt to the cavity walls.

The null hypotheses were rejected, since the gap formation and the bond strength at the cavity was significantly affected by the placement of the fiber. The result of the current study has demonstrated the protective effect of the fiber for the deep dentin floor. However, it is noteworthy that the placement of the fiber is time-consuming and technique sensitive. Therefore, a clinical technique should be optimized since the success of the fiber placement depends on the full integration and impregnation of the fiber into the resin.

5. Conclusion

The interfacial integrity of the bulk-fill composite to cavity floor improved with the placement of a fiber-reinforced increment at the base of the deep preparation. The fiber-reinforced increment may act as a shrinkage stress breaker and protect the bonded interface in deep dentin.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at https://doi.org/10.1016/j.dental.2020.01.007.

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