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ORIGINAL ARTICLE

Repair bond strength of bulk-fill composites: influence of different primers and direction of debonding stress

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ABSTRACT

Background: The purpose of this *in vitro* study was to evaluate the effect of different adhesion primers on the repair bond strength of bulk-fill resin composite and short-term hydrolytic stability of the repair interface before and after accelerated aging. In addition, direction of debonding stress was examined.

Materials and methods: Bulk-fill substrates were aged in water for 14 days at 37 °C. Smooth resin composite surfaces were prepared for the substrates with a superfine grinding paper (FEPA #500, #1200, #2000). Test specimens were produced by attaching bulk-fill composite to the substrate surfaces, using three different primer/bonding systems. Specimens were aged 24 h at 37 °C in water, or thermal cycled (5–55 °C/5,000 cycles). Subsequently, shear bond strength and micro-tensile bond strength were evaluated. In total there were 60 specimens for the shear bond strength and 60 specimens for the micro-tensile bond strength measurements (30 stored in water 24 h, 30 thermal cycled, $n = 10$ in each primer/bonding mode).

Results: The mean shear bond strength was 9.1–13.1 MPa after 24 h water storage and 6.9–10.7 MPa after thermal cycling. The mean micro-tensile bond strength was 28.7–45.8 MPa after 24 h water storage and 22.7–37.9 MPa after thermal cycling.

Conclusion: The Ceramic primer (silane containing) seems to perform better than the three-step etch and rinse adhesive or the Composite primer. Shear-type stress had an adverse effect on the repair bond strength of bulk-fill resin composites.

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Bonding; bulk-fill; shear bond strength; micro-tensile bond strength

1. Introduction

Restorative treatments cause a large workload for dentists partly due to the high prevalence of untreated caries. Toothwear is another common reason for healthy tooth substance loss [1–4]. Resin composites are primarily used as a dental restorative material, which consists of fillers and for instance bisphenol-A-glycidyl dimethacrylate (Bis-GMA) and other dimethacrylate monomers. In these materials, polymers form polymer networks by additional polymerization [5,6]. Bulk-fill resin composites were developed to simplify and speed up the placement of large posterior fillings. The material can be placed in increments of 4–5 mm [7,8]. However clinical research is still limited concerning their use in posterior lesions.

As with other restorations, resin composite fillings have finite survival time, replacement of dental filling usually leads to an increase in cavity size, and destruction of healthy tooth substance [9]. Nowadays, repair of defective resin composite restorations is considered a valid treatment option. For this purpose, the new filling material will be bonded to the old one and mechanical retention is often used as an aid. Repair bond strength describes the adhesion zone intensity between these two interfaces. Repair of resin composite fillings' preserves healthy tooth tissue, improves the prognosis of the filling's, and reduces operation time, and costs [10–12]. Few studies have been published concerning the repair bond strength of aged bulk-fill resin composites [13,14].

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Adhesion-promoting primers are used in resin composite repairs [15]. These can theoretically have an influence on the polymer matrix or inorganic filler part of the bonding substrate. To begin with, dissolution or swelling of the polymer surface is desired to take place but dissolution of the polymer matrix requires a non-cross-linked interpenetrating polymer network (IPN). Typically cross-linked polymer matrix of dental resin composites cannot be properly dissolved by conventional dental adhesives or primers [16–20]. On average, half of the surface of the resin composite consists of exposed inorganic filler. To achieve adequate bond strength between old inorganic fillers and the new repair composite, the intention is to promote siloxane-based covalent bonding with the help of silane coupling agents [21]. Silane-promoted adhesion may be over time prone to hydrolysis, which potentially weakens the interfacial bond [22,23]. The following primer/bonding solutions were included in this study to evaluate if these can aid chemical bonding. Adper™ Scotchbond™ Multi-Purpose Primer + Adhesive (3 M ESPE, St. Paul, MN, USA) is a three-step etch-and-rinse system [24], while G-premio bond (GC, Tokyo, Japan) is a universal adhesive [25], and Composite primer (GC, Tokyo, Japan) is especially indicated for composite-to-composite bonding [26]. Ceramic primer II (GC, Tokyo, Japan) contains additional silane indicated for repair bonding [27]. Further, Filtek One Bulk Fill (3 M ESPE, St. Paul, MN, USA) is a packable nano-filled bulk-fill resin composite [28]. Resin composite bonding can be measured in several ways like shear or tensile bond strength tests in which the direction of stress is different. In general, it́s acknowledged that tensile-stress testing results in higher values when compared to shear-stress testing [13,22,23,29,30]. Few studies have compared the effect of loading (stress) direction for interfacial bond strength of composite repairs. In certain clinical conditions load is shear in type whereas in others it can be tensile stress or a combination.

The present study aimed to evaluate the effect of three different adhesion primers on the repair bond strength of the commonly used bulk-fill resin composite. In addition, direction of debonding stress was examined using shear and micro-tensile bond strength tests (SBS, μTBS) before and after accelerated ageing. The secondary aim encompassed the evaluation of the fracture zone using stereomicroscopy. The hypothesis was that there would be no difference in the repair bond strength when using different adhesion primers nor when using different directions of debonding stress.

2. Materials and methods

The materials used in this study and their composition are given in Table 1. The Filtek One Bulk Fill substrates for the SBS test were prepared by placing the material in plastic tubes (7.5 mm diameter, 4 mm height). The tubes were coated with petrolatum on the inner surface and placed on a Mylar strip on a table. The bulk-fill composite was inserted in one increment and light-cured 20 s from the top of the mould using a Demi Ultra led curing lamp (Kerr, Orange, CA, USA) with an irradiance of 1236 mW/cm^2 (measured by Norwegian Radiation Protection Authorities, Österaas, Norway). This lamp was used throughout the study. The bulk-fill substrates for the μTBS test were prepared in the same way as described above (plastic tubes 7.5 mm diameter, 6 mm height). The substrates were light-cured 20 s from the top and bottom sites. The distance of the light tip from the composite surface was 1 mm in all studied samples. The manufacturer's guidelines were followed in all phases. After the curing stage, the plastic moulds were carefully removed. The bulk-fill substrates were stored in 37 °C distilled water immediately after production for 14 days.

After water storage, the bulk-fill substrates for SBS test were embedded in rounded acrylic supports (ClaroCit, Struers, Copenhagen, Denmark). The substrates were wet ground with grinding paper (FEPA #500, #1200, #2000, Struers, Copenhagen, Denmark) under running water. The same grinding method was used for the μTBS substrates but without acrylic support. Approximately 0.5 mm composite was ground off at the top to achieve a flat smooth surface so that the actual effect of primers on the bond strength could be assessed. SEM images of the polished surfaces are presented in Figure 1. The flat surfaces to be repaired were cleaned with Top Dent 38% etch gel (Pulpdent, Watertown, MA, USA) for 15 s and rinsed under water for 15 s. Three different primers and bonding modes were studied: (1) Adper Scotchbond Multi-Purpose Primer + Adhesive, (2) Composite primer, (3) Ceramic primer II + G-premio bond (Table 1). The primers and bonding solutions were applied according to the manufacturers' specifications regarding drying, rubbing of the surface, air blow, time, and light curing.

The primer/bonding combinations were tested after 24 h water storage at 37 °C or after artificial ageing by thermal cycling (TC) i.e. 5.000 cycles at temperatures ranging from 5 to 55 °C (20 s at each temperature, intermediate time of 2–3 s). Further, the intention was also to investigate both SBS and μTBS for all modes.

Table 1. Materials used in the study and their composition.

Material	Manufacturer	Composition	Lot no	Expiration Date
Filtek one bulk fill restorative, shade A3 (Shear and microtensile)	3M ESPE, St. Paul, MN, USA	Fillers: silica, zirconia, ytterbium trifluoride Organic matrix: AUDMA, UDMA, 1, 12-dodecane-DMA	NF27545 NF40402, NF26085, NF40908	2025-02-19 2025-05-26
Etch gel	Pulpdent®, Watertown, MA, USA	38% phosphoric acid	210907	2023-09-07
Adper™ Scotchbond™ Multi-Purpose Primer	3M ESPE, St. Paul, MN, USA	HEMA, water, copolymer of polyalcenoic acid	NF25422	2024-07-30
Adper Scotchbond multi-purpose adhesive	3M ESPE, St. Paul, MN, USA	HEMA, BisGMA, Triphenylantimony	NF25422	2024-07-30
Composite primer	GC, Tokyo, Japan	HEMA, UDMA, Tetrahydrofurfuryl methacrylate	2101201	2023-01-19
Ceramic primer II	GC, Tokyo, Japan	MDP, 2.2′-Ethylene dioxyethyl dimethacrylate, (1-methyl Ethylidene) bis [4,1-phenylenoxy (2-hydroxy-3,1-propanedyl)] bis methactylatelate	2203101	2024-03-09
G-Premio bond	GC, Tokyo, Japan	4-MET, 10-MDP, MDTP	2205131	2024-05-12

AUDMA: Aromatic Urethane-dimethacrylate; UDMA: Urethane-dimethacrylate; DMA: Dimethylacetamide; HEMA: Hydroxyethyl methacrylate; BisGMA: Bisphenol A-glycidyl methacrylate; MET: Methacryloxyethyl trimellitic acid; MDP: Methacryloyloxydecyl dihydrogen phosphate; MDTP: Methacryloyloxydecyl dihydrogen thiophosphate.

Specimens used for SBS testing were made by applying repair bulk-fill composite (Filtek One Bulk Fill) cylinders (3 mm diameter, 3 mm height) to the substrate surface by using a device as described in ISO/TS 11405:2003 [31]. The new composite was light-cured 20 s. In total, 60 specimens for the SBS test were prepared (30 stored in water 24 h, 30 TC, $n = 10$ in each primer/bonding group). The sample size was based on a previous study by Staxrud [32]. Specimens were placed in 37 °C distilled water for 24 h before testing.

Specimens used for μTBS testing were made by applying repair bulk-fill composite (Filtek One Bulk Fill) using the same diameter plastic tubes as used for the bulk-fill substrates. The mould was placed on top of the specimen and a 6 mm high repair bulk-fill composite cylinder was placed in one increment. Excess material was cleaned from the margins with a spatula and the composite was light cured 20 s on top and 40 s around the adhesive site. The mould was removed carefully, and specimens were placed in 37 °C distilled water for 24 h. The bulk-fill composite specimens for the μTBS test were sectioned into approximately 1.1 × 1.1 mm square-shaped sticks with a precision cutting machine (Secotom-60, Struers, Copenhagen, Denmark) under water cooling with a sharp diamond blade perpendicular to the bonding surface. Approximately 10 test sticks were prepared from each specimen. After cutting, the sticks were cleaned ultrasonically in distilled water for 10 s. The sticks were then examined under a stereo microscope at a magnification of 40x to ensure that the surfaces were intact. Only specimens without defects were accepted. The width and thickness of each test specimen were measured to the nearest 0.01 mm using a calibrated digital caliber (Mitutoyo Co, Kawasaki, Japan). In total, 60 samples were prepared for μTBS measurements (30 stored in water 24 h, 30 TC, $n = 10$ in each primer/bonding group).

2.1. Shear bond strength

SBS was evaluated after 24 h water storage and after TC. The SBS test was performed according to the ISO/TS 11405:2003 [31] standard. The specimens were fixed in a brass cylinder using a Teflon ring and a small set screw. Then the specimens were fixed in a mounting jig and a shear force was applied parallel to the flat-prepared bonding site at a speed of 1 mm/min using a universal testing machine (Instron 1121, Instron, High Wycombe, Bucks, UK) until failure. The bond strength was calculated by dividing the maximum load at failure (N) with the bonding area (mm^2). The results were recorded in megapascal (MPa).

2.2. Micro-tensile bond strength

μTBS was evaluated after 24 h water storage and after TC. The square-shaped test sticks were attached to the bond test machine as described by Eliasson et al. [33,34]. The sticks were fixed 2 mm inside extension screws (ELRA AS, Oslo, Norway) with cyanoacrylate glue (Locktide 435, Hankel Norden, Gothenburg,

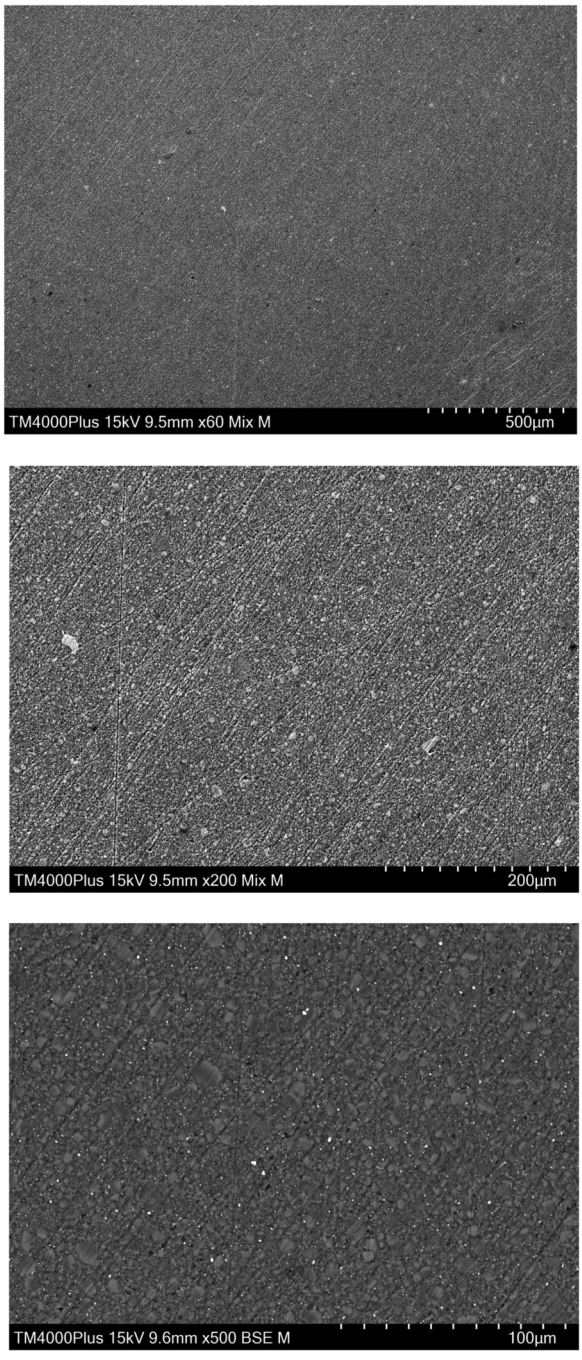

Figure 1. SEM images of the bulk-fill substrate surfaces that were polished with grinding paper (FEPA #500, #1200, #2000). Magnification ×60, ×200, ×500.

Sweden). The specimens were placed in a particularly designed fitting mould to ensure correct alignment along the long axis of the stick. After this, the sticks were fixed from the other end into a calibrated universal testing machine (Lloyd Instruments LTD, Model LRX, Fareham, England) with particularly developed steel cords. The cords applied tensile forces perpendicularly to the specimen's bonding site at a speed of 1 mm/min until fracture. The bond strength was calculated by dividing the maximum load at failure (N) with the bonding area (mm^2). The results were recorded in megapascal (MPa). After the SBS and μTBS tests, the specimens were inspected with a stereomicroscope (American Optical, Buffalo, NY) at 40x magnification to determine if the fracture was located in an adhesive or in a cohesive zone.

2.3. Statistical methods

A Kolmogorov-Smirnov test was used to examine the normal distribution of the bond strength measurements. The normally distributed data were presented as mean values and standard deviations for each experimental group. The results were evaluated by analysis of variance (ANOVA). Tukey's post-hoc test was used. The statistically significant level was set at $p < 0.05$. The statistical analyses were performed using SPSS Statistics, v 27.0.1.0 (IBM, New York, USA).

3. Results

The SBS and μTBS values are presented in Table 2 and fracture sites are presented in Table 3. The highest and equal SBS values (13.1 MPa) after 24 h water storage was achieved with Adper Scotchbond Multi-Purpose Primer + Adhesive and Ceramic primer II + G-premio bond. After TC the highest SBS was with Ceramic primer II + G-premio bond (10.7 MPa). However, there were no statistically significant differences between the primer/bonding groups after the SBS test. After the SBS test, all fractures were in the adhesive zone. The highest μTBS value (45.8 MPa) after 24 h water storage was achieved with Ceramic primer II + G-premio bond. After TC the highest μTBS was with Ceramic primer II + G-premio bond (37.9 MPa), which was statistically significantly higher compared to the other primer groups. All specimens in the Composite primer group failed after TC. More specifically in this group the bulk-fill composite to bulk-fill composite repair bond broke and failed while the specimens were cut to sticks.

4. Discussion

In the present study, flat smooth resin composite surfaces were prepared for the bulk-fill substrates to see the actual effect of primers on the repair bond strength with only a little mechanical interlocking. Based on the results Ceramic primer performed better than the three-step etch and rinse adhesive or Composite primer. The repair bond interface was

Table 2. The shear and micro-tensile bond strengths after 24 h water storage or after thermal cycling for the different adhesion primers used for bulk-fill to bulk-fill repair.

		Adper™ Scotchbond™ Multi-Purpose Primer + Adhesive	Composite primer	Ceramic primer II + G-Premio bond	p
Shear bond strength					
	Water storage 24 h	13.1 (4.4)	9.1 (4.6)	13.1 (5.2)	NS
	Thermal cycling	9.5 (4.2)	6.9 (4.1)	10.7 (6.1)	NS
Micro-tensile bond strength					
	Water storage 24 h	28.7 (5.5)	29.2 (15.9)	45.8 (11.7)	NS
	Thermal cycling	22.7 (13.3)	–	37.9 (9.8)	*

Data are shown as means ± SD. The statistical differences between the adhesion primers combinations were evaluated by analysis of variance (ANOVA). NS $p > 0.005$. * Statistically significant difference between different primers, $p = 0.004$.

Table 3. The fracture sites after the shear and micro-tensile bond strength tests.

		Adper Scotchbond multi-purpose primer + adhesive	Composite primer	Ceramic primer II + G-Premio bond
Shear bond strength				
	Cohesive fracture after 24 h water storage	0	0	0
	Adhesive fracture after 24 h water storage	10	10	10
	Cohesive fracture after thermal cycling	0	0	0
	Adhesive fracture after thermal cycling	10	10	10
Micro-tensile bond strength				
	Cohesive fracture after 24 h water storage	2	6	2
	Adhesive fracture after 24 h water storage	8	4	8
	Cohesive fracture after thermal cycling	0		2
	Adhesive fracture after thermal cycling	10		8

more prone to shear-type stress compared to tensile stress. Thus, the null hypothesis was rejected. All the micro-tensile bond strength values were higher compared to shear bond strength values. This indicates that shear-type stress is adverse to the repair bond strength of bulk-fill resin composites. The highest repair bond strength was achieved with Ceramic primer II + G-premio bond after 24 h water storage or TC based on the SBS and μTBS evaluations. Ceramic primer II contains MDP monomer whereas Adper Scotchbond and Composite primer include HEMA monomer. MDP monomer is known for its beneficial adhesion and hydrophobic properties [35]. Hydrophobic properties are due to molecule structure where a long spacer chain separates the functional groups. This prevents water absorption, which may weaken bonding properties [36]. Good repair bond strength potential of 10-MDP monomer is also found in earlier studies concerning the same subject [22,23]. It can theoretically bond to oxide groups in the fillers of the composite being repaired, which might explain the results in the present study [37]. In addition, 10-MDP monomer can react with possible unreacted C = C double bonds in the resin matrix. This might be essential for the repair bond strength although the probability for reaction of the unreacted C = C bonds of the substrate with free radical monomer units of the adhesive of the repair resin composite is low [23,38]. Further, Ceramic primer differs from the other adhesives in this study by containing a silane [27]. Silanes can theoretically form siloxane bonds to filler particles in the old composite to enable chemical bonding between fillers and new resin matrix. The silane agent might also improve the repair bond strength by wetting as it enables direct contact between different materials, which is essential for a strong bond [39].

After 24 h water storage six of the Composite primer group fractures were located in the cohesive area, which is more than in other groups. The cohesive fractures might result from weak points in the surface, as discerned through observations derived from stereomicroscopic imagery. Cohesive fractures exhibit marginal significance on the outcomes, especially when positioned in close proximity to the adhesion zone. Overall, the number of cohesive fractures was low among all studied samples, which indicates that the results describe the actual repair bond strength

between old and new filling [34]. Further, all the Composite primer group specimens failed after TC. It seems that the Composite primer including HEMA monomer without adhesive application as per the manufacturer's instructions could be prone for hydrolysis.

In an earlier study the SBS for Filtek One Bulk Fill substrates repaired with the same material was 18.8 MPa when a self-etch adhesive was used and 19.1 MPa when a totally etched was used [22]. In our study the substrate surfaces were polished to reduce mechanical interlocking, which probably explains the lower SBS values. In other studies repair bond strength values were approximately on the same level as our results. For example [23], and [30] evaluated μTBS of bulk-fill composite repaired with a nanohybrid resin composite. Without aging, repair bond strength was 26.1–46.8 MPa and 28.5–43.5 MPa after TC. The difference in the results is probably explained by different resin composites and adhesives used. Further, in other studies μTBS repair bond strength for Filtek^{TM} One Bulk Fill was 26.3.5–47.2 MPa. The substrates were treated with different adhesion protocols including etching, silane, etch and rinse adhesive, universal adhesive, and mechanically abraded with a bur, which probably explains the higher bond strength values compared to our results [13,29].

In the present study both SBS and μTBS tests were used to simulate the loading direction for interfacial bonding. This can be seen as a strength as most previous studies concerning the same subject used only μTBS testing. The bulk-fill substrates were stored in 37 °C distilled water immediately after production for 14 days to complete the polymerization reaction. The study design was chosen to simulate the repair interface of resin composite. Also, little is known about the effect of chemical bonding on the repair bond strength of bulk-fill composites. In most earlier studies mechanical interlocking was used, which includes abrasion with a bur, air abrasion, and sandblasting [13,23,29,30]. The SBS test is widely used for adhesive bond strength testing although it poorly simulates repeated clinical load in the mouth [40]. Due to this the μTBS test was additionally performed in our study. In this test method, pretesting failures can be a problem [41]. For example, fixing the specimen during sectioning can be difficult, cutting the specimen with a diamond blade may weaken the adhesive zone, and gluing of sticks can cause differences between the sticks. Further, the tubes used for specimen preparation were coated with petrolatum on the inner surface which may have contaminated the bulk-fill material. These shortcomings should be considered when comparing the results. Further, the number of samples was relatively low which is why additional investigations are needed to confirm the results.

5. Conclusions

The Ceramic primer (silane containing) seems to perform better than the three-step etch and rinse adhesive or the Composite primer. Shear type stress had an adverse effect on the repair bond strength of bulk-fill resin composites.

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Disclosure statement

No potential conflict of interest was reported by the author(s)

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