Original and Repair Bulk Fracture Resistance of Particle Filler and Short Fiber–Reinforced Composites

J Bijelic-Donova • S Uctasli • PK Vallittu • LVJ Lassila

Clinical Relevance

Longevity of repaired direct composite restorations may be improved by including a short E-glass fiber-reinforced composite with a semi-interpenetrating network matrix as the substrate material in bilayered restorations.

SUMMARY

Objective: This study aimed to evaluate the original (OR) and repair (RR) fracture resistance of a semi-interpenetrating polymer network (semi-IPN)-based short fiber-reinforced composite compared to dimethacrylate-based composite materials by means of the V-notch test.

Methods and Materials: Circular specimens $(5\times2$ mm) with a centrally machined 90° V-

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shaped notch were prepared. Four bulk fill (Filtek Bulk Fill, Venus Bulk Fill, TetricEvo Ceram Bulk Fill, SDR), three microfilled hybrid (GC-Anterior, GC-Posterior, Z250), one nanofilled (SupremeXTE), and two short fiber-reinforced (Alert, everX Posterior) composites were selected. EverX Posterior was the semi-IPN material. Specimens (n=12/group) were either dry or water stored for 7 and 30 days, respectively, at 37°C and then loaded in two-point load until fracture. One-half of each tested specimen was used for the repair procedure. Repairing surfaces were diamond-bur ground, etched, and treated with silane containing universal adhesive (Scotchbond Universal) before repair.

Results: Three-way analysis of variance revealed a significant statistical difference between the groups (p<0.05). The fracture resistance of dry-stored groups was greater than that of water-stored groups. The highest OR was observed for dry-stored Alert (23.4 N/mm), which significantly deteriorated in water (17.4 N/mm) (p<0.05). The highest RR was observed for everX Posterior (20.0 N/mm), which did not deteriorate in water significantly (19.0 N/mm) (p>0.05). The everX Posterior preserved the specimens' integrity at the final fracture load

(ductile fracture), whereas all other materials fractured into two halves at the interface (adhesive failure).

Conclusions: The only material that provided enhanced repair strength that was close to the original cohesive strength of the material was everX Posterior. The endurance of repaired restorations can be improved by using semi-IPN-based filling material.

INTRODUCTION

Replacement, repair, and refurbishment are types of interventions employed for restoring defective dental restorations. The least invasive treatment is refurbishment, which is used when an existing restoration has to be reshaped or refinished or has overhangs that need to be removed. A repair is more conservative than replacement; a repair is used to restore a defective part of a restoration, whereas replacement involves removal of an entire restoration.¹

The bonding between the old and the new composite in repair is important. Although repair is a simple intervention, there is no consensus on the best course of treatment of the repairing substrate surface prior to repair. The most commonly used means for surface treatment of the repairing composite are macro- or micromechanical roughening with a bur, grinding paper, sandblasting with aluminum oxide particles or silica-coated particles, and etching with hydrofluoric acid.² Some of these, such as grinding paper or hydrofluoric acid, are unfeasible or unsafe methods for direct intraoral repair. In addition, the safety of sandblasting has been also questioned due to the aerosol contamination of the operating room.3 Instead, clinically friendlier techniques are etching with phosphoric acid or grinding with diamond burs. 4-7 The last one was shown to provide equally good⁶ or improved⁴ repair bond strength compared to sandblasting with aluminum oxide particles. Surface roughening is followed by an application of an intermediate layer, which could be a silane agent alone or in combination with a bonding agent,^{5,8} an unfilled or a filled bonding agent^{5,6,9,10} or a low-viscosity flowable composite. 4,5 Usually, mechanical or chemical treatment used alone is ineffective in reestablishing the original strength of the material, but used together they improve the repair strength. 1,10 Lately, it has also been observed that bonding agent used alone as an intermediary layer aids the repair bond, which is an important finding because a clinician would normally apply an adhesive layer to the whole cavity.

In order to establish a durable repair, both mechanical and chemical bonding must be achieved. The type of the composite to be repaired is crucially important, as different resin matrices (cross-linked, linear, or a combination of both) have different chemical and physical properties. 11,12 The bonding of a new composite to an aged thermoset matrix (dimethacrylate) in repairs is poor. This is because thermoset matrices are highly cross-linked and nondissolvable by monomers. 12-14 On the other hand, thermoplastic matrices (eg polymethylmethacrylates) have good reparability potential due to their linear chains. The combination of both phases, thermoset (cross-linked) and thermoplastic (linear), in one matrix system is known as a semi-interpenetrating polymer network (semi-IPN). ¹³ This matrix type has good reparability potential due to the presence of polymethylmethacrylate chains, which are easily dissolvable with monomers that have solubility parameters close to it. 13,15 Semi-IPN has been described for continuous fiber-reinforced composites (FRCs) and has been proven to be superior to cross-linked resin matrix in FRC repairs 11,12 because fresh monomers diffuse significantly deeper into semi-IPN than into cross-linked FRC. 14,16 Currently. however, only one semi-IPN-based composite material for filling applications is available on the market. This restorative material contains short fibers 0.85 to 1.09 mm in length, ¹⁷ and it therefore represents a semi-IPN-based short fiber-reinforced composite (SFRC). According to the fiber aspect ratio theory, 18-20 this material could be classified as highaspect-ratio SFRC (short fiber length on a millimeter scale). This is important because only low-aspectratio SFRCs (short fiber length on a micrometer scale) have been presented on the market thus far. 21

The semi-IPN-based high-aspect-ratio SFRC is commercially known as everX Posterior (EXP, GC Corp, Tokyo, Japan). It has been shown that the use of this SFRC obtains cohesive fractures between immediately incrementally placed layers of composite, even on removal of the oxygen inhibition layer.²² This property could be advantageous in composite repairs, when this composite is the repairing substrate, that is, the underlying material. Furthermore, the mechanical and structural characterization of EXP revealed that protruding fiber ends and bridging fibers contribute to the fracture toughness of the material.¹⁷ Based on these earlier findings, it could be hypothesized that the presence of both semi-IPN matrix and protruding fibers could enhance the repair strength of the EXP, even though the silane is removed from the fiber surface due to grinding. Consequently, an

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Material (Manufacturer)	Code and Lot Number	Type of Composite	Resin Matrix
Filtek Z250 (3M ESPE, Seefeld, Germany)	FZ250 N565032	Microhybrid	Bis-GMA, Bis-EMA, UDMA, TEGDMA
Filtek Supreme XTE (3M ESPE)	FS N649930	Nanofilled	Bis-GMA, Bis-EMA, UDMA, TEGDMA
G-aenial Anterior (GC Corp, Tokyo, Japan)	GA 1501151	Microfilled hybrid	UDMA, dimethacrylate comonomers (Bis-GMA free)
G-aenial Posterior (GC)	GP 1301161	Microfilled hybrid	UDMA, dimethacrylate comonomers (Bis-GMA free)
Smart Dentin Replacement (Dentsply, York, PA, USA)	SDR 151020	Flowable bulk fill	Modified UDMA, TEGDMA, EBPDMA
Filtek Bulk Fill (3M ESPE)	FBF N600679	Flowable nanofilled bulk fill	Bis-GMA, Bis-EMA, UDMA, procrylate
Venus Bulk Fill (Heraeus Kulzer, GmbH, Hanau, Germany)	VBF 010030	Flowable nanohybrid bulk fill	UDMA, EBPDMA
Tetric Evo Ceram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein)	TBF R72542	High viscosity (sculptable) nanohybrid bulk fill	Bis-GMA, Bis-EMA, UDMA
everX Posterior (GC)	EXP 1309111	High-aspect-ratio (millimeter- scale) SFRC ^a	Bis-GMA, TEGDMA, PMMA
Alert (Jeneric/Pentron, Nallingford, CT, USA)	Alert 3762763	Low-aspect-ratio (micrometer-scale) SFRC ^b	Bis-GMA, UDMA, TEGDMA, THFMA,
Scotchbond Universal (3M ESPE)	SU 589527	Filled phosphate dimethacrylate— based universal adhesive resin	Bis-GMA, HEMA, MDP, polyethylene glycol, water, initiator, silane

Abbreviations: Bis-GMA, bisphenol-A-glycidyl dimethacrylate; bis-EMA, bisphenol-A-dyethoxy dimethacrylate; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; EBPDMA, ethoxylated bisphenol A dimethacrylate, PMMA: polymethylmethacrylate; THFMA, tetrahydrofurfuryl-2-methacrylate; HEMA, hydroxyethylmethacrylate; MDP, methacryloyloxi-decyl-dihydrogen-phosphate.

objective in this study was to investigate the repair properties of the semi-IPN-based SFRC (EXP) in comparison to dimethacrylate-based composites. In addition, one low-aspect-ratio dimethacrylate-based SFRC (Alert, Jeneric/Pentron, Wallingford, CT, USA) was evaluated (Table 1). The original and repair strengths were evaluated using the V-notch test, which measures the bulk fracture resistance, known also as bulk toughness or torque to failure. This method was originally developed in 1995 by Uctasli and others. 23 The advantages of the method are that specimen size is approximate to the size of a dental restoration and that the test setup enables the assessment of stress in a dental restoration during occlusion. Indeed, the V-notch section resembles the fissure of a posterior tooth, whereas the roller fixed in the testing device resembles the antagonist.²³

METHODS AND MATERIALS

Ten commercially available composites were investigated, including four bulk fill composites, three microfilled hybrid composites, one nanofilled com-

posite, one low-aspect-ratio SFRC, and one high-aspect-ratio SFRC. The high-aspect-ratio SFRC was the semi-IPN-based composite material (EXP). The other materials were dimethacrylate-based composites, and all were shade A3. The restorative materials, their abbreviations, and codes are presented in Table 1.

Specimens (n=12/group) were prepared in a circular Teflon mold, 5 mm in diameter and 2 mm in thickness, with a centrally machined V-shaped notch. The angle of the central notch was accurately 90°. Original and repair fracture resistance (OR and RR, respectively) was evaluated by this test.

Monolithic Specimens (OR)

The material was packed into the V-shaped mold, pressed with a Mylar strip, and covered with a glass plate in order to exude excess material. Prior to polymerization, the glass plate was removed, and the specimen was light polymerized against the Mylar strip in intimate contact with the light-curing unit tip

^a High-aspect-ratio SFRC: short fiber length on a millimeter scale

b Low-aspect-ratio SFRC: short fiber length on a micrometer-scale.

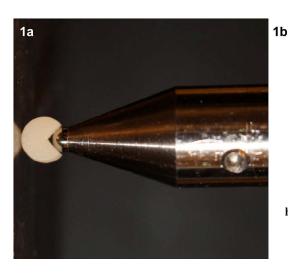
Table 1: Extended.			
Material (Manufacturer)	Filler Type	Filler, wt%/vol%	
Filtek Z250 (3M ESPE, Seefeld, Germany)	Zirconia/ silica: 0.01-3.5 μm	78/60	
Filtek Supreme XTE (3M ESPE)	Aggregated zirconia/silica cluster: 0.6-1.4 μm and nonagglomerated/nonaggregated silica filler: 20 nm	78.5/59.5	
G-aenial Anterior (GC Corp, Tokyo, Japan)	Prepolymerized: silica and strontium and lanthanoid fluoride containing filler 16-17 μm; silica filler >100 nm and fumed silica filler <100 nm	80/63	
G-aenial Posterior (GC)	Prepolymerized: silica and strontium and lanthanoid fluoride containing filler 16-17 µm; fluoro-alumino-silicate >100 nm and fumed silica filler <100 nm	81/65	
Smart Dentin Replacement (Dentsply, York, PA, USA)	Barium-alumino-fluoro-boro-silicate and strontium-alumino-fluoro- silicate: 4.2 μm	68/44	
Filtek Bulk Fill (3M ESPE)	Zirconia/silica: 0.01-3.5 μm Ytterbium trifluoride: 0.1-5.0 μm	64.5/42.5	
Venus Bulk Fill (Heraeus Kulzer, GmbH, Hanau, Germany)	Barium-alumino-fluoro-silicate and ytterbium trifluoride, silica: 0.02-5 μm	65/38	
Tetric Evo Ceram Bulk Fill (Ivoclar Vivadent, Schaan, Liechtenstein)	Barium-alumino-silica, spherical mixed oxide, prepolymer filler (ytterbium trifluoride, glass filler, and monomer 17 wt%)	80 (with 17% prepolymers)/60	
everX Posterior (GC)	E-glass fiber (0.3-1.9 mm) Barium-boro-silicate	Filler: 74.2/53.6 E-glass: 8.6/7.2	
Alert (Jeneric/Pentron, Wallingford, CT, USA)	E-glass fiber (60-80 μm) Barium-boro-alumino-silicate: 0.7 μm	Total, filler and fiber: 84/70	
Scotchbond Universal (3M ESPE)	Colloidal silica nanofiller Vitrebond copolymer: fluoro-alumino-silicate glass powder based	Not applicable (trade secret)	

(0-mm distance) for 20 seconds using a light-polymerizing unit (Elipar S10, 3M ESPE, Seefeld, Germany), having a 10-mm-diameter tip, an output intensity of 1600 mW/cm², and a wavelength range between 430 and 480 nm. On removal from the mold, specimens were additionally polymerized from the top side for 20 seconds and were then stored at 37°C either dry for seven days or in water for 30 days before testing. The thickness of each specimen was measured prior to testing with a digital caliper with an accuracy of ± 0.002 mm (Mitutoyo Corp, Tokyo, Japan).

A fracture test was conducted in a universal material testing machine (Lloyd model LRX, Lloyd Instruments Ltd, Fareham, UK). The external force was applied with a cylindrical roller 3 mm in diameter in two-point opening (mode I) tensile load at a crosshead speed of 1.0 mm/min until failure. The cylindrical roller provided a two-point contact at the V-shaped notch, enabling equal distribution of the load with the purpose of breaking the specimens into two halves (Figure 1a and 1b).

Repaired Specimens (RR)

After testing, one-half of the fractured specimen was used for repair. The fracture surface of each half specimen was ground with a fine-grit diamond bur (40-μm mean grit size), etched with 37% phosphoric acid (Scotchbond Universal Etchant, 3M ESPE) for 15 seconds, washed with water spray for 15 seconds, and dried from a ~5-mm distance. A bonding agent (Scotchbond Universal adhesive, 3M ESPE) was then rubbed onto the surface using a microbrush applicator for 15 seconds, gently air-dried, and light cured for 20 seconds. The specimen was then returned to the mold, and new material was bonded to it by filling the vacant end with the same material used for fabrication of the original specimen in one increment. The repair specimens were thus a "sandwich" of old and new composite. In other words, the repairing substrate was the old composite, which was previously aged and tested. Preparation and light-curing steps were otherwise the same as for the monolithic specimens. Repaired specimens were made within seven days after testing the monolithic specimens. Following repair, storage mediums and storage times for the repaired specimens were identical with those of the original specimens; dry specimens were exposed to seven days of dry storage, and water-stored specimens were subjected to water storage for 30 days, both at 37°C. Specimens were E236 Operative Dentistry



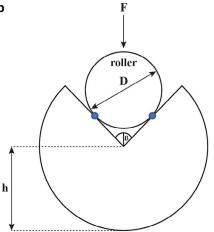


Figure 1. Assembly of the V-shape test. (a): Specimen positioned under roller during testing. (b): Diagram. F, load applied; D, diameter of the roller (3 mm); h, height from notch to base; "\$\beta\$, half angle of the right-angled notch; solid circles, places of two-point contact.

fractured as described earlier for the monolithic specimens.

OR and RR values were derived in accordance with the following equation:

$$T = FD 0.71/2 th$$
,

where T is the fracture resistance in newton millimeters (N/mm), F is the applied load in newtons (N), D is the roller diameter (3 mm), t is the specimen thickness (2 mm), and h is the height from notch to base (2.5 mm). When the specimen's notch is 90°, its half angle is $\emptyset = 45^\circ$ and $\sin 45^\circ = 0.71$, and this was the value used in the previous equation.

Fracture pattern analysis and scanning electron microscopy (SEM) investigations were conducted for EXP specimens, which were the only ones that did not break into two halves. The crack interface of three specimens (n=3) was analyzed. The specimens were ground wet (Stuers LabPol-21, Stuers A/S, Copenhagen, Denmark) with silicon carbide papers of decreasing abrasiveness (1000, 1200, and 4000 grit) and gold sputter coated prior to SEM examination.

Statistical Analysis

The data were statistically analyzed with SPSS version 23 (SPSS, IBM Corp, Armonk, NY, USA). Three-way analysis of variance (ANOVA) followed by the Tukey post hoc test (α =0.05) was used to determine if the fracture resistance was different for monolithic and repaired specimens (OR and RR), for the materials investigated (10 materials), and for the type of storage medium (dry or water). Material, storage type, and fracture resistance category (OR and RR) were independent variables, and fracture

resistance (OR, RR results) was the dependent variable. Two-way ANOVAs followed by the Tukey post hoc test (α =0.05) were also conducted to evaluate the interactions between the material type and the storage medium individually for monolithic and repaired specimens (ie, for OR and RR separately).

RESULTS

OR was higher compared to RR for both dry- and water-stored specimens. Three-way ANOVA showed statistically significant interactions for factors "fracture resistance category (OR or RR)" (p<0.05), "material type," and "fracture resistance category" (p=0.00003) and for "storage medium," "material type," and "fracture resistance category" (p=0.047) but not for "storage" and "fracture resistance category" (p=0.075). However, there was a concern that the effect that each type of material had on the fracture resistance category (OR or RR) might have been different for dry and water storage media. Two-way ANOVA confirmed this, showing that the interaction between the material type and the storage medium was significant for OR (p=0.014) but not for RR (p=0.332). Statistical differences across the fracture resistance values and storage mediums for each material are presented in Figure 2.

Fracture resistance deteriorated in water. This was significant for the majority of materials in the OR category (p<0.05) but insignificant for the majority of materials in the RR category (p>0.05). The highest OR value was observed for dry-stored Alert specimens (23.4 N/mm), which significantly deteriorated in water (17.4 N/mm) (p<0.05). The highest RR value was observed for EXP specimens (20.0 N/mm), which did not significantly deteriorated

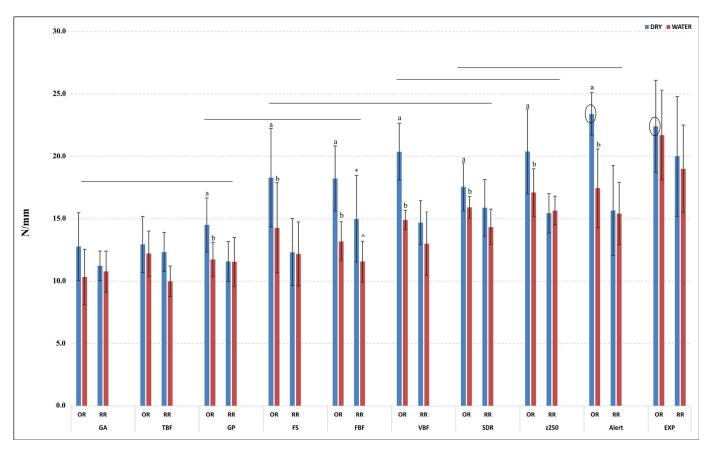


Figure 2. Graphs showing original (OR) and repaired fracture resistance (RR) values of investigated materials. Horizontal lines above columns indicate groups that were statistically similar (p>0.05). For each material analyzed separately, small letters show the statistical difference between dry and water OR values, whereas symbols show the statistical difference between dry and water RR values. Circled areas show that for the SFRC materials, only dry groups were statistically similar (p>0.05). GA. G-aenial Anterior; GP, G-aenial Posterior; TBF, Tetric Evo Ceram Bulk Fill; FS, Filtek Supreme XTE; FBF, Filtek Bulk Fill; VBF, Venus Bulk Fill; SDR, Smart Dentin Replacement; Z250, Filtek Z250; EXP, everX Posterior.

rate in water (19.0 N/mm) (p>0.05). Alert initially performed similar to EXP but significantly weakened on storage in water (p<0.05) and on repair (p<0.05), behaving in these cases as a microhybrid PFC (FZ250). Furthermore, EXP showed statistically higher OR and RR values (p<0.05) compared to all PFC materials investigated in this study.

Among the repair specimens, all specimens, except EXP, broke into two halves at the interface side. This adhesive mode of failure was a sign that the weakest link was the repair interface. Following testing, the EXP specimens (100%) were not detached. This was classified as a cohesive failure, that is, ductile fracture.

SEM analyses were conducted for unbroken EXP specimens. SEM revealed protruding fibers at the interface (pulled out or broken) and bridging fibers near the notch, where the fracture initiated (Figure

3a), and bridging fibers at the middle and end part of the fracture line (Figure 3b,c).

DISCUSSION

To date, no optimal or universal repair protocol has been vet established for composite repairs.²⁴ Principally all factors—the monomer, 12 the composite type, 10,24 and the intermediary agent type 25—influence the repair strength. Generally accepted is that the macro- or micromechanical roughening is superior to chemical surface treatment. 4,6,10 Nonetheless, it has been also observed that an intermediate bonding agent layer alone^{1,8} or in combination with a silane agent⁸ could be sufficient in repairs as well. Currently, surface grinding with a diamond bur followed by an application of a bonding agent⁴ or phosphoric acid etching followed by both silane and bonding agent application²⁴ are recommended as the safest, most efficient repair techniques. These methods combine effectiveness and safety and are E238 Operative Dentistry



Figure 3. (a-c): SEM micrographs showing typical fracture line segments. (a): SEM micrograph (500×) showing protruding fibers at the interface (red arrows) and bridging fibers (white arrows) at the beginning of the fracture line (near the notch). (b): SEM micrograph (500×) showing bridging fibers (white arrows) at the middle part of the fracture line. (c): SEM micrograph (500×) showing bridging fibers (white arrows) at the end part of the arrested fracture line.

what all clinicians would most likely perform due to the simplicity of the technique. For these reasons, etching followed by an application of silane-containing universal adhesive of the previously diamondroughened repair surface was the repair technique of choice for the purposes of the present study.

When repairing PFCs, the substrate is composed of thermoset matrix type and fillers. Fresh composite layers bond via the oxygen inhibition layer, but bonding of a new composite to aged thermoset matrix (dimethacrylate) is weak because of the cross-linked nature of the thermoset matrix. 12-14 If, however, the matrix is the semi-IPN type, the bonding possibilities during repair improve, and this is due to the secondary IPN bonding, which is possible only for the semi-IPNs. 13 More precisely, the principle of this type of bonding lies in the fact that the linear phase (polymethylmethacrylate) of the semi-IPN structure can be dissolved with monomers that have solubility parameters close to it. Examples of those are monomer solutions containing hydroxyethylmethacrylate (HEMA) or methylmethacrylate in combination with high-molecular-weight dimethacrylates. 14-16 The new resin swells and diffuses into the "old" noncross-linked (linear) polymer matrix. On polymerization of the newly applied material, an adhesive type of bond, namely, the secondary IPN, is established. 13,15 In other words, the secondary IPN happens via dissolution of the semi-IPN substrate. In the present investigation, the secondary IPN was possible only for the EXP because it contains short Eglass fiber fillers embedded in a dimethacrylate resin matrix modified with polymethylmethacrylate linear chains. Moreover, the short fibers (0.85 to 1.09 mm in length¹⁷) protrude from the (substrate) surface and interlock with the newly applied composite layer, thus allowing a mechanical type of bonding for EXP. Indeed, SEM evaluation revealed protruding fibers at the fracture surface in this (Figure 3) and a previous study. 17 Consequently, because of the semi-IPN-promoted secondary IPN (adhesive bond) and the mechanical interlock (mechanical bond), a reliable repair was obtained, as observed in RR values. The results concur with findings of previous studies that illustrated the secondary IPN bonding mechanism in repairing aged semi-IPN-based continuous FRC¹¹ and showed that repair bond increases if resin matrix is semi-IPN. 12 To clarify, the difference between semi-IPN-based continuous FRCs and SFRCs is that continuous FRCs are used for reinforcing fixed dental appliances, whereas SFRC (EXP) is a filling material. Furthermore, protruding fibers were earlier shown to be responsible for the improved immediate shear bond strength and cohesive failure mode of EXP in the absence of an oxygen inhibition layer, 22 which was observed also in the present study (RR values). Indeed, protruding fibers allowed the ductile fracture types seen in this study, which is a sign of a durable adhesion. On the other hand, the resin matrix in Alert is thermoset, and for this reason the adhesive

type of secondary IPN bond is not achievable in repairs for this SFRC material.

In the absence of dissolvable matrix, the basis of PFCs' repair is roughening the repairing surface. Indeed, roughening the surface enlarges the bonding surface area and allows the keying with the newly applied material via the intermediate agent. This agent could be silane followed by adhesive application, 8,9,24 universal adhesive that contains silane, 8,9,26,27 or adhesive alone. 8,9 The intermediate agent wets the surface, absorbs to the roughened filler particle, and levels the surface irregularities by seeping into them.²⁸ Thus, it allows the interlocking mechanism between the adhesive and the repairing substrate and facilitates the diffusion of the new composite to the macromechanical porosities. It should be noted, however, that roughening alone is not an adequate surface treatment because it removes the silane layer from the exposed fillers, and the newly applied composite layer inadequately wets the surface.²⁹ In other words, adhesive treatment of the roughened surface enhances the repair.28

It should be noted that Alert has crushed fibers and that EXP has short fibers, which both already themselves enlarge the bonding surface. In addition, the areas between protruding fibers are gap-like and promote the interlocking mechanism between two successive composite layers. Consequently, short fibers behave as microretentive elements and aid better adhesion. Hence, it follows that SFRCs are naturally retentive, and in repairs two retentive forms could be achieved: *natural* microretention provided by the short fibers and macroretention developed by roughening the surface.

Several different types of PFCs were investigated in the current study. Although monomer types in various PFC brands are similar, there are some differences in the monomer concentrations; filler type and filler content are also different for different composite brands (Table 1). There is some evidence, however, that the presence of silica filler particles within the composite might be the key factor affecting the repair, which was related to the reactivity of the silica particles.²⁴ All PFCs investigated were silica based, which could explain the similar values. However, based on the accompanying filler type, materials in this study could be divided into 1) PFCs, either barium-alumino-fluoro-silicate based or zirconia-silica based, and 2) short-FRCs, both E-glass based. Thus, the differences could be explained by the variations in filler composition, content, and size. Namely, it has been shown that the etching effect depends on the filler particle composition; if the fillers are easily displaced by the etchant, as is the case with barium fillers, the possibility for silanization in re-restoration weakens because the etched surface loses fillers and matrix dominates.³⁰ Likewise, an efficient silanization is difficult also for prepolymerized fillers, but this is a direct consequence of their manufacturing technique.³¹ The poor impregnation of the prepolymerized filler (by the resin matrix) leads to reduced mechanical properties,³¹ and this was also observed in the present investigation. Namely, prepolymerized fillers containing composites (in this study GC-Anterior, GC-Posterior, and TetricEvo Ceram Bulk Fill), with ~ 63 vol% fillers, had similar values among them that were significantly lower than the other composites (Figure 2). This could also be attributed to the critical filler level (50 to 60 vol%) above which any further addition of filler could diminish the mechanical properties of the material.³² Alert initially performed similar to EXP but significantly deteriorated on storage in water and on repair when behaving as microhybrid PFC (FZ250). This finding could indicate that the micrometer-scale crushed fibers acted as micrometer particulate fillers.

Furthermore, only one type of coupling agent was used in the present study. Scotchbond Universal adhesive is a prehydrolyzed silane containing adhesive with an acidic phosphate monomer 10-methacryloyloxi-decyl-dihydrogen-phosphate (MDP) and HEMA; that is, it is a phosphate-dimethacrylatebased silane-containing adhesive. It has been shown to be a good silanizer, which creates a consistent surface²⁷ and improves the repair strength of aged PFC substrates. 26,27 Clinically, its use could reduce the clinical steps because it eliminates the need for using separate silane and adhesive during the composite repair.³³ Furthermore, because of its low viscosity, the bonding agent could flow into the irregularities created during surface grinding and could improve bonding due to macroretention. Nonetheless, stability and effectiveness of the silane within this adhesive have been of some concern.³⁴ Namely, the low pH of the Scotchbond Universal adhesive might initiate premature hydrolysis and dehydration condensation, characteristics that make its silane unstable.³⁴ In the present investigation, the use of this agent led to an average RR of 84%, which was somewhat lower for water-stored specimens. A possible explanation could be that both acidic solution and water storage caused the hydrolysis of the silane agent that consequently led to E240 Operative Dentistry

degradation. The aim of an intermediate layer is to make a transition interphase between the old and the new composite, but there is no consensus on whether any type of intermediate layer could be used for improving the repair interface²⁸ or whether the repair is bonding system dependent.²⁵ There was no counterpart for comparison included in this study design (group without the use of the agent), which makes further conclusions related to the use of the bonding agent inconclusive. Thus, it remains open whether another bonding agent, separate silane, or application of flowable composite would have improved the RR.

Mechanical properties of composites usually deteriorate in water, ³⁵ which was also the case in this study. Both OR and RR deteriorated in water, but this reduction was more significant for OR than for RR. This finding could be due to the incomplete water saturation during short-term storage in water. Repaired specimens were prepared by adding new material to aged substrate, which contained absorbed water. This caused a significant decrease of RR and could be explained with the fact that repaired specimens were previously stressed and already aged. Clinically, this could mean that a repaired restoration is weaker because it was previously subjected to forces and aging conditions encountered in the oral cavity.

The interaction between the material type and the storage medium was significant for OR but not for RR. The only material that provided enhanced and almost comparable OR and RR was the semi-IPNbased SFRC (EXP). This finding indicates that while the original cohesive strength of the restorative materials is material dependent, the type of dimethacrylate-based substrate is not as important for the repair procedure. First, all repairs for dimethacrylate-based composite materials were considerably lower than their original counterparts, and, second, all failed at the repair interface. Consequently, the findings of this study reinforce the theory that the repair of dimethacrylate-based composite materials cannot be reliably achieved, whereas the semi-IPN matrix enhances the repair strength. The interaction between the material type and the storage medium was also significant only for OR. The absence of any interaction on repair could be attributed to the previously mentioned fact that the repairing substrate was already stressed and aged.

Fracture resistance (torque to failure) measured by the V-shape test is a rarely evaluated material characteristic, ^{23,36-38} particularly for FRCs. ³⁸ The method was developed in 1995 by Uctasli and others,

and, as the authors have stated, it resembles a clinical situation of stressing dental restoration in occlusion.²³ The V-notch is the fissure and is the side where stress concentrates, the sides of the notch are the cuspal inclines where the two-point contact occurs, and the cylindrical roller is the antagonist that generates the stress. ²³ The role of the notch is to allow a sharp flaw in the material and facilitate crack initiation and propagation, which makes fracture resistance measurement more accurate. Therefore, creation of the notch is the most relevant step in this test type. The authors tested a few notch angulations and specimen dimensions and concluded that a circular 2 × 5-mm specimen with 90° notch angle approaches cavity size and is suitable for assessing the fracture resistance behavior of restorative materials.²³ To date, by this method, a number of PFCs were tested^{23,36,37} but only one continuous FRC. 38 In the later study, unidirectional FRC was placed in different positions at the notch end, simulating placements beneath the cusp, at the fissure base or gingival side of a pontic.38 The authors found it difficult to place the fibers in the right position in such a small mold cavity but showed that the fiber position was the most important factor affecting bulk fracture resistance. The results of the present study are consistent with the finding that fiber reinforcement enabled ductile fracture and impeded crack propagation.³⁸ Any difficulties with SFRC application were not experienced, however. Furthermore, accordant results were found for the mutual PFCs investigated earlier;36,37 the small variances could be due to the different storage periods and media.

One limitation of the present study was that only one surface treatment was used. However, since other methods have not been proven to significantly recover the composite repair bond, ³⁹ this disadvantage of the current study design is relative.

This *in vitro* study presents substantial potential of the semi-IPN-based SFRC (EXP) to improve the longevity of repaired restorations. However, only clinical studies of repaired EXP restorations could actually show the effectiveness of the semi-IPN and the millimeter-scale fibers in achieving this.

CONCLUSIONS

Within the limitations of the present investigation, it could be concluded that diamond-bur roughening followed by phosphoric acid etching and an application of silane-containing bonding agent is a simple treatment procedure and reliable repair method because the fracture resistance of the repaired specimens was, on average, 84% of the original bulk fracture resistance.

The presence of millimeter-scale short E-glass fibers in the semi-IPN matrix enables durable bonding in composite repairs (absent an oxygen inhibition layer), and factors contributing to this are 1) the natural microretentive feature of the short fibers, 2) interface reinforcement by protruding and bridging fibers, and 3) semi-IPN-mediated secondary IPN. This is important because ductile failure and durable bonding are signs of clinical reliability of the adhesion. In other words, the longevity of the repaired restoration could be enhanced in this way.

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of approval of the University of Turku, Institute of Dentistry.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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