Repair bond strength of restorative resin composite applied to fiber-reinforced composite substrate

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Tezvergil A, Lassila LVJ, Yli-Urpo A, Vallittu PK. Repair bond strength of restorative resin composite applied to fiber-reinforced composite substrate. Acta Odontol Scand 2004;62:51–60. Oslo. ISSN 0001-6357.

Delamination or fracture of composite veneers can occur as a result of improper design of the fiber-reinforced composite (FRC) framework. This in vitro study tested the repair bond strength of restorative composite to aged FRC. The substrate was multiphase polymer matrix FRC (everStick) aged by boiling for 8 h and storing at 37°C in water for 6 weeks. The aged substrate surfaces were wet-ground flat with 1200-grit silicon carbide paper and subjected randomly to 5 different surface treatments: 1) An adhesion primer (Composite Activator) and resin (CA), 2) Silane (EspeSil) and resin (SIL-MP), 3) Silane, adhesive primer, and resin (Clearfil Repair) (CF), 4) Air particle-abrading (CoJet), silane, and resin (CJ-SIL-MP), 5) Resin (Scotchbond Multipurpose Resin) only as control (MP). Restorative composite resin (Z250) was added to the substrate in 2 mm layer increments and light-cured. Subsequently, every surface treatment group was divided into 2 subgroups of 12 specimens each. The specimens were either 48 h water-stored or thermocycled (6000 × 5–55°C). The shear bond strengths of composite resin to FRC were measured at a crosshead speed of 1.0 mm/min. The data were analyzed by ANOVA for factors 'treatment type' and 'storage condition'; Tukey's post-hoc tests and Weibull analysis were performed. ANOVA showed a significant difference as a function of surface treatment ($P < 0.05$) and storage condition ($P < 0.05$). The CJ-SIL-MP group showed highest bond strength and Weibull modulus after thermocycling. Repair of multiphase polymer matrix FRC may show reliable bond strength when silane treatment is used along with air-particle abrading. □ *Air-abrading; fiber-reinforced composite; repair; shear bond strength; silane*

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The development of fiber-reinforced composite (FRC) materials and technologies offers minimally invasive clinical alternatives for various kinds of fixed partial dentures (FPD) (1–3). Good esthetics and metal-free frameworks with high fracture strength can be obtained using FRC (4, 5).

High quality dental FRC products have been preimpregnated either with light polymerizable monomers (Targis Vectris®, FiberKor®) or with a porous linear polymer (Stick®) containing about 45% volume of fibers in the fiber-rich phase of the end product (4, 5). A recent introduction is a new FRC material composed of densely packed silanated E-glass fibers in a light-curing gel matrix (everStick®) (6). The matrix contains poly(methylmethacrylate) (PMMA) chains in a bisphenol A-glycidyl dimethacrylate (bisGMA) matrix encapsulated by a PMMA layer to improve handling as well as bonding properties.

Bonding between the FRC framework and veneering particulate filler composite is achieved by chemical and physical means (7, 8). When the fibers are impregnated with light-curing monomers that are di- or multifunctional methacrylates, the polymer matrix of FRC can only be bonded to the veneering composite by means of mechanical interlocking or free radical polymerization of the unreacted functional groups of the matrix of FRC. As the number of unreacted double bonds and free radical activity decreases after polymerization, the possibility to obtain free radical polymerization diminishes over time.

Another mechanism for adhering veneering composite resin to FRC is based on interdiffusion bonding of resin monomers to the FRC substrate, which results in the formation of a semi-interpenetrating polymer network (SIPN) (9). It has been shown that by using a suitable resin it is possible to bring about diffusion of resin monomers into the polymer matrix of FRC (10), hence sufficient bond strength values of veneering composite even to an aged FRC framework (11) can be obtained. Interdiffusion bonding can occur more quickly in a linear polymer matrix than in a cross-linked polymer matrix. This was the basis for the development of FRCs such as Stick and everStick, both of which contain PMMA in the multiphase polymer matrix.

However, with the increase in the fiber volume fraction of high strength FRCs, the possibility of obtaining bonding through interdiffusion is decreased because of the lowered volume percentage of the multiphase polymer matrix. This might be problematic, especially during the repair of FRC materials.

Air-particle abrasion may be another method by which adhesion strength can be increased as a result of mechanical interlocking. It has been reported that this method

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DOI 10.1080/00016350310008210

Table 1. Materials used in this study

Brand	Code	Manufacturer	Lot no.	Chemical composition
EverStick	ES	StickTech, Turku, Finland	201.0723.ES025	PMMA, bisGMA, E-glass
Z250	Z25O	3 M Dental Products, St. Paul, MN, USA	OHR	bisGMA,UDMA, bisEMA
3 M Scotchbond Multipurpose Adhesive	MP	3 M Dental Products, St. Paul, MN, USA	20011115	bisGMA, HEMA
Composite Activator	CA	Bisco, Dormage, USA	010635	Methylmethacrylate, butylmethacrylate
Clearfil Repair	CF	Kuraray Co., Ltd., Osaka, Japan	41156	A: MDP, HEMA, Water, B: MDP, bisGMA, HEMA, Hydrophobic dimethacrylate, silanated colloidal silica C:Bis-Phenol-A-polyethoxydimethacrylate, MPTS
CoJet Sand	CJ	3 M Dental Products, St. Paul, MN, USA	0005	Aluminum oxide ~95% Coated with silica ~5%
Espesil	SIL	3 M Dental Products, St. Paul, MN, USA	140046	Ethyl alcohol 99% MPTS 1%

BisGMA = Bisphenol A-glycidyl dimethacrylate; UDMA = Diurethane dimethacrylate; bisEMA = Bisphenol A polyethylene glycol diether dimethacrylate; HEMA = Hydroxyethylmethacrylate; MDP = 10-methacryloyloxydecyl dihydrogen phosphate; MPTS = 3-methacryloxypropyl trimethoxysilane; A = SE Bond Primer; B = SE Bond Adhesive; C = Porcelain Bond Activator.

provides a repair strength of 60% of cohesive bonding values (7, 12). Several reports have been published on the repair strength of FRC with the cross-linked polymer matrix using one of these methods (8–12). However, no studies have been undertaken evaluating the repair strength of composite resin to multiphase polymer matrix FRC.

The aim of the current in vitro study was to investigate repair bond strength of restorative composite to FRC with multiphase polymer matrix. In addition, the Weibull distribution of the data was evaluated in order to compute bond failure probabilities as a function of treatment method and storage condition.

Materials and methods

The materials, manufacturers, and lot numbers used in this study are listed in Table 1. Unidirectional E-glass fiber reinforcement preimpregnated with bisGMA and PMMA (everStick) was used as a substrate for the adherence of restorative composite resin (Z250). One-hundred-and-twenty substrate specimens were fabricated by placing unpolymerized FRC material into the retentive cavity (8 × 6 × 4 mm) in acrylic resin, surrounded by a stainless steel cylinder. The substrate was light-polymerized with a hand-held light-curing device (Optilux-501, Kerr, CT, USA) for 40 s. The wavelength of the unit was between 380 and 520 nm and light intensity was 720 mW/cm^2 measured with the light-curing device's internal radiometer. To simulate aging of FRC, the substrate specimens were first water-stored at 37°C for 48 h, then boiled in water for 8 h, and again water-stored at 37°C for 6 weeks. The aged substrate surfaces of the specimens were wet-ground flat with 1200-grit (FEPA) silicon carbide grinding paper. The specimens were randomly subjected to 5 different surface treatments (Table 2). Repair of the aged substrate surface with an adhesive resin was used as control. Particulate filler composite Z250 was then applied in 2 mm increments to the substrate surface using a

Table 2. Surface treatment protocol for FRC substrates before application of restorative composite resin

Groups	n/group	Interphase layer	Storage condition*
MP (CONTROL)	12/12	MP was applied to the surface and left to react for 1 min and light cured for 10 s.	Water storage/ Thermocycled
CA	12/12	2 layers CA were applied on ES surface using a brush and left to react for 1 min. MP was then applied as described.	Water storage/ Thermocycled
CF	12/12	A + C mixed and applied to the surface for 5 s and dried with mild air flow. B applied to the surface and light cured for 10 s.	Water storage/ Thermocycled
SIL-MP	12/12	SIL was applied on ES surface and dried with mild air blow. MP was then applied as described.	Water storage/ Thermocycled
CJ-SIL-MP	12/12	ES surface air-abraded with CJ for 10 s at 10 mm distance and was cleaned with water spray. SIL and MP were applied as described.	Water storage/ Thermocycled

* After application of restorative composite.

translucent polyethylene mold with a diameter of 3.6 mm, and each increment was polymerized with the light-curing device for 40 s.

Subsequently, every surface treatment group was divided into 2 subgroups according to the storage conditions, with 12 specimens in each subgroup. The subgroups were stored in deionized grade 3 water at 37°C for either 48 h or 24 h, and then additionally thermocycled for 6000 cycles between (5 ± 2)°C and (55 ± 2)°C with a dwell time of 30 s and a transfer time of 5 s. Twenty-four hours after thermocycling, a shear bond test was performed using a universal testing machine (model LRX, Lloyd Instruments, Fareham, England). The specimens were secured in a mounting jig (Bencor Multi-T shear assembly, Danville Engineering Inc., San Ramon, Calif., USA) with the shearing rod against and parallel to the flat prepared bonding sites. A circular edge blade created the shear type load positioned over the FRC-restorative composite interface using a universal testing machine (LRX, Lloyd Instruments, Fareham, UK) at a crosshead speed of 1.0 mm/min. Substrates were standardized so that the fibers were oriented in all specimens parallel to the shear force. Fracture surfaces of the specimens were examined visually and selected specimens from each group were evaluated with a scanning electron microscope (SEM) (JSM-5500, Jeol Ltd, Tokyo, Japan) to determine their failure region. In addition, the surfaces were analyzed using the energy dispersive X-ray spectroscopy (SEM-EDS) system in order to measure the area percentage of the fibers at the surface using image analysis software (Spirit, Princeton Gamma-Tech Inc., N.J., USA). Analyses were based on the difference in the elemental composition of glass fibers compared to that of polymer matrix.

Shear bond strength values for all groups were analyzed with analysis of variance (ANOVA) for the factors ‘treatment type’ and ‘storage condition’; for interactions, one-way ANOVA and Tukey’s multiple comparisons post hoc analysis were employed at a significance level of $P < 0.05$ with statistical software SPSS 10.0 (Statistical Package for Social Science, SPSS Inc., Chicago, Ill., USA).

Weibull analysis is a statistical approach that has been used to evaluate the fracture process in dental materials (13–15). The shear bond strength data for the groups of 12 identically size specimens were ranked in ascending order and a Weibull analysis was performed on the resultant data. The basic form of the Weibull distribution is shown as:

$$P_f = 1 - \exp(-((\sigma - \sigma_u)/\sigma_0))^m \qquad (1)$$

where m = Weibull modulus (also known as a shape factor), a constant, which determines the slope of the distribution function and characterizes the spread of the failure data with respect to the σ (= strength) axis. σ_o = characteristic strength (i.e. the stress level at which 63% of the specimens have failed) and σ_u = theoretical failure stress at which the failure probability approaches zero and is known as the threshold stress (MPa). P_f is the probability of failure, which varies from 0 to 1, and median ranks for estimated fracture probability is calculated from the formula (also known as Benard’s approximation P_{fe}) (13):

$$P_{fe} = \frac{n - 0.3}{N + 0.4} \qquad (2)$$

where N is the total number of specimens in the group and n is the ranking number of the specimens ranked in ascending order.

The correlation coefficient r was calculated with linear regression from logarithmic transformations, and r-values above 0.9 were considered statistically acceptable (13–15). The stress at 10% failure probability was calculated for each group, and survival probability curves were examined in an attempt to assess the distribution of shear bond strengths.

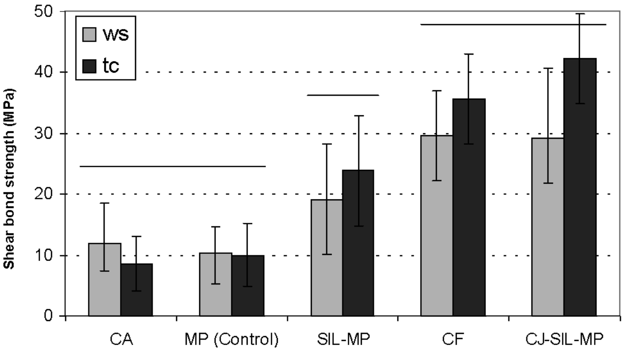

Fig. 1. The means and standard deviations of shear bond strength in relation to different substrate treatments and storage conditions. Vertical lines represent standard deviations and horizontal lines above surface treatment columns do not differ from each other (WS = water-stored and TC = thermocycled). For group codes, see Table 2.

Results

The results of the shear bond strength test are shown in Fig. 1. The highest bond strength values (42.2 MPa) were achieved with the CJ-SIL-MP treated surface after thermocycling. The CA and MP groups showed similar bond strength values at the 10 MPa range, but these were low compared to the other groups. Two-way ANOVA revealed that there was a significant difference in the

Table 3. Effect of surface treatment and storage condition on the shear bond strength of specimens compared by two-way ANOVA

Factors	Sum of squares	d.f.	Mean square	F	*P* value
Surface treatment	13,994	4	3498.5	56.04	0.000
Storage	530	1	530.5	8.50	0.004
Surface treatment*storage	964	4	241	3.86	0.006

Table 4. Weibull analysis of test groups

Group	Storage condition	Characteristic strength = σ_0	Weibull modulus = m	Correlation r-coefficient	Stress for 10% probability of failure
CA	Water-stored	13.48	2.09	0.92	4.6
CA	Thermocycled	11.68	0.64	0.87	3.7
CF	Water-stored	32.63	4.09	0.95	18.8
CF	Thermocycled	39.08	4.46	0.94	23.6
MP	Water-stored	11.74	2.61	0.94	5.0
MP	Thermocycled	12.28	2.42	0.97	4.9
SIL-MP	Water-stored	22.31	1.61	0.96	5.6
SIL-MP	Thermocycled	27.26	2.39	0.94	10.7
CJ-SIL-MP	Water-stored	35.13	2.91	0.96	16.2
CJ-SIL-MP	Thermocycled	45.39	6.04	0.97	31.3

results as a function of surface treatment ($P < 0.05$) and storage condition ($P < 0.05$). The interaction between these factors was also significant ($P < 0.05$) (Table 3).

Bond strength of the silanated groups showed a modest increase after thermocycling. The increase in the SIL-MP and CF group was not statistically significant ($P > 0.05$), whereas it was statistically significant for CJ-SIL-MP ($P < 0.05$).

Bond strength of CF showed slightly lower values (36.5 MPa) compared to CJ-SIL-MP, but the difference was not statistically significant. A silane layer between Z250 and ES increased bond strength (23.8 MPa) but was not as effective as the CF group.

The Weibull distribution parameters σ_o, m, correlation coefficient, and 10% failure probability are listed in Table 4. Compared to the MP and CA surface treatments, the Weibull characteristic strength (σ_o), at which 63% of specimens failed, increased significantly after silane was applied in groups 3, 4, and 5 (Figs 2 and 3). The m parameter ranged from 0.64 to 6.04 (corresponding to standard deviation), indicating a wide distribution. For all groups except the CA thermocycled group, the linear correlation of the data (Table 4) was acceptable ($r = 0.92$–0.97). The thermocycled CA group showed an r-value lower than 0.9, as 2 samples did not fit the distribution.

The 10% probability of failure of the control group MP was 5.0 MPa for water storage and 4.9 MPa for the thermocycled groups. CA showed lower values than the control, whereas the other groups showed higher values (Table 4).

Visual analysis of fracture surfaces revealed that CA and MP produced adhesion failures (Fig. 4), whereas fracture surfaces of CF and CJ-SIL-MP showed mostly cohesion failures. SIL-MP produced a mixture of adhesion and

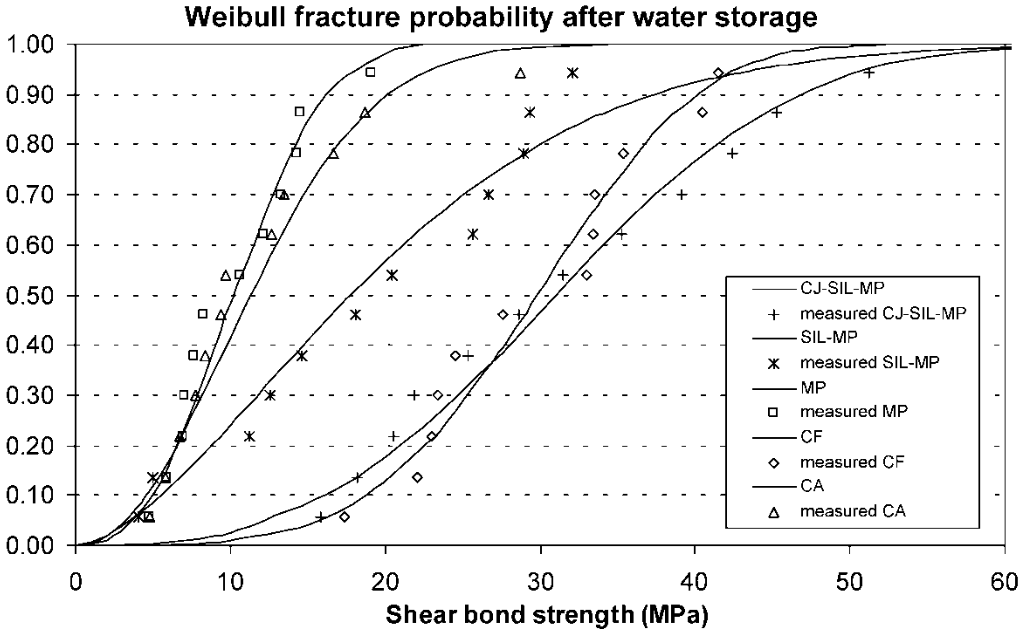

Fig. 2. Cumulative fracture probability curves for water-stored specimens.

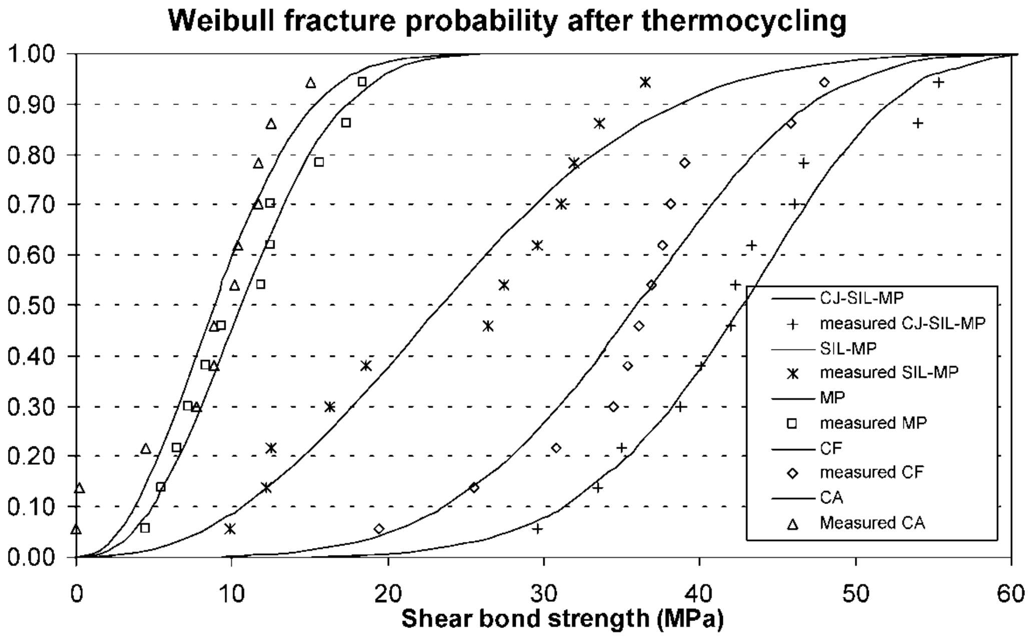

Fig. 3. Cumulative fracture probability curves for thermocycled specimens.

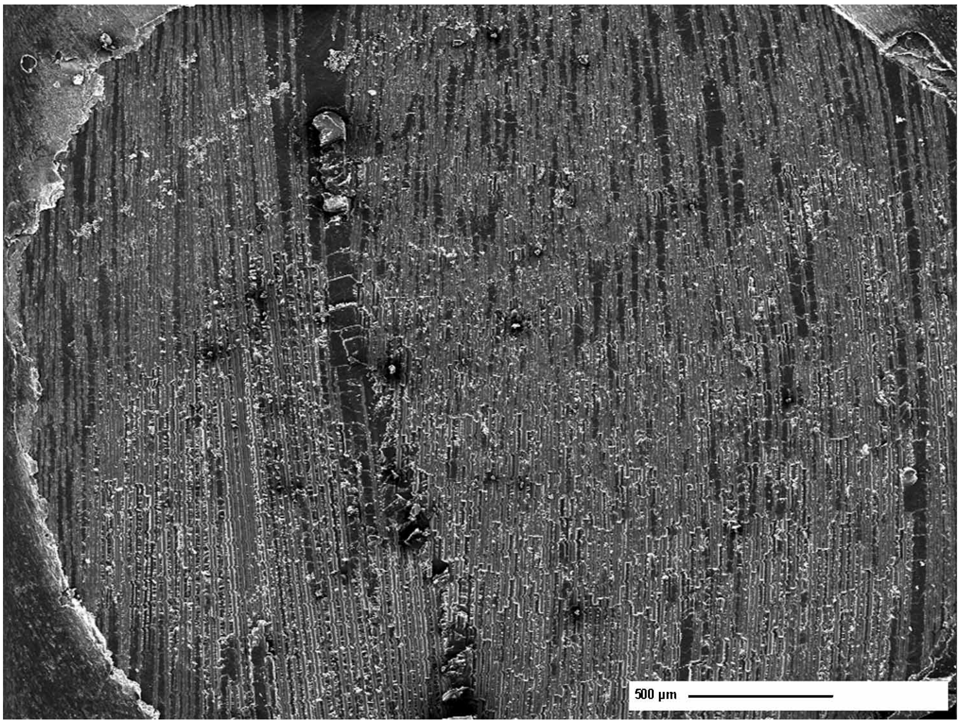

Fig. 4. SEM micrographs of fracture surface of MP adhesive group (control). Original magnification ×35, bar = 500 μm.

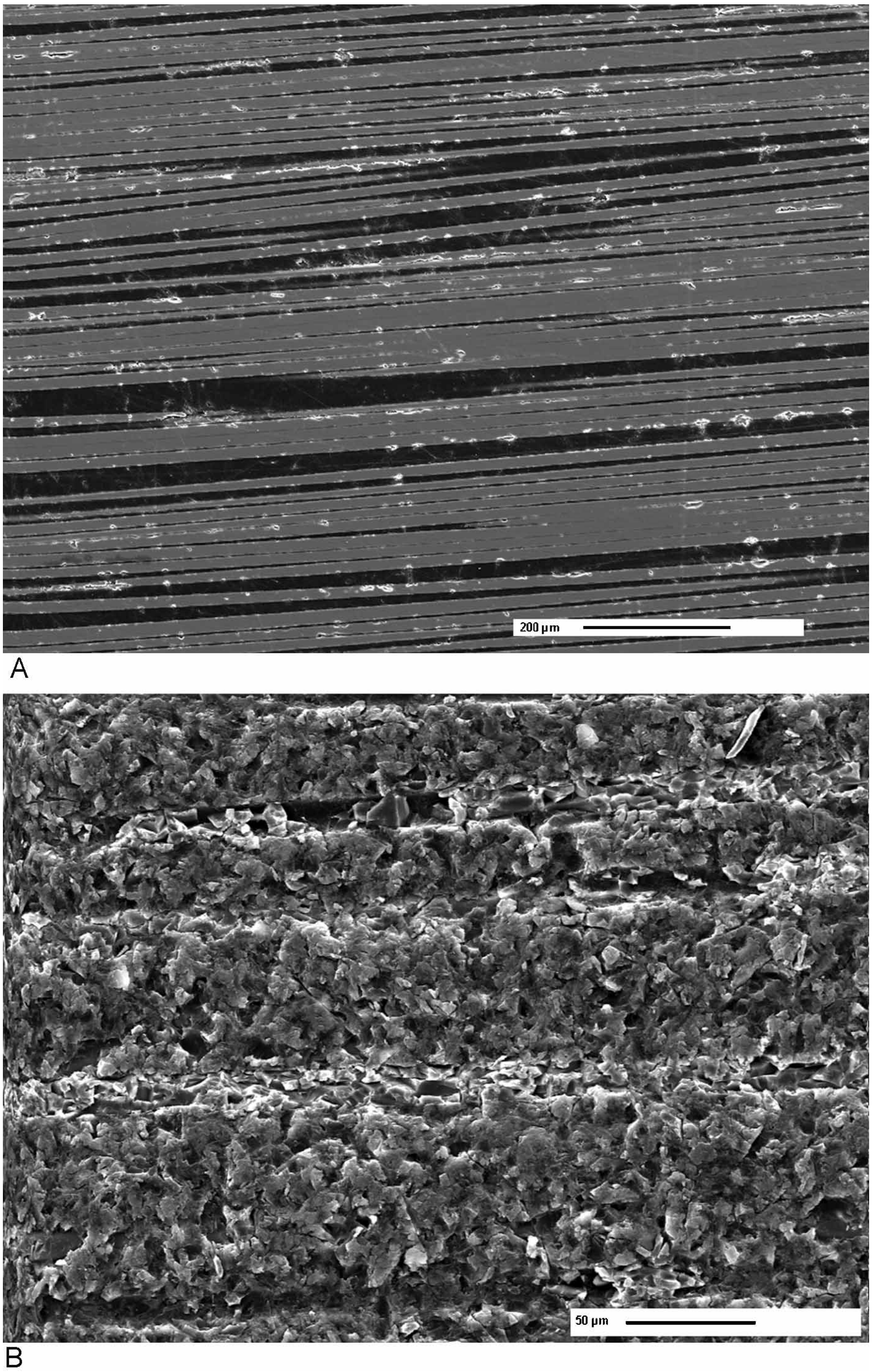

Fig. 5. A. SEM micrographs of the ground surface of everStick (substrate) using 1200-grit paper. Original magnification ×100, bar = 200 µm. B. SEM micrographs of the surface of everStick (substrate) after CoJet treatment. Original magnification ×350, bar = 50 µm.

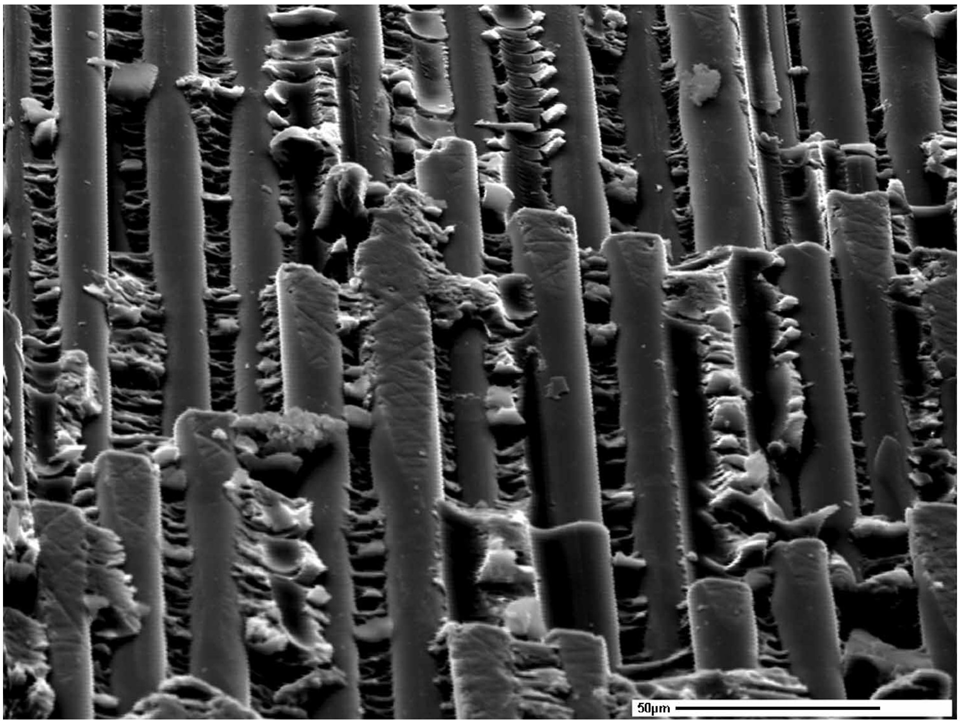

Fig. 6. SEM micrographs of fracture surface of ClearFil (CF) group. Original magnification ×500, bar = 50 µm.

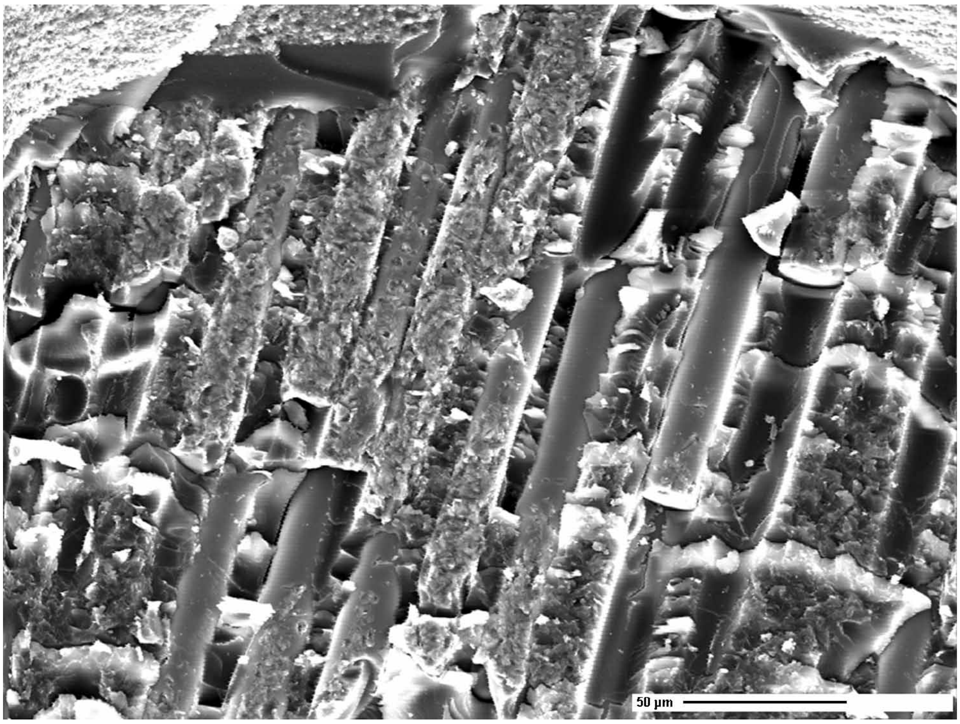

Fig. 7. SEM micrographs from fracture surface of CJ-SIL-MP group. Original magnification ×400, bar = 50 µm.

cohesion failures. SEM micrographs confirmed the visual analysis. SEM micrographs of ground surfaces showed a smooth surface pattern (Fig. 5a), whereas the CJ air-abraded surface showed increased surface roughness and even some deterioration of fibers on the FRC substrate (Fig. 5b).

CF applied to the smooth-ground substrate surface produced cohesion failures either on the substrate or adherend (Fig. 6); these were similar to the cohesion failures produced by the CJ-SIL-MP groups. Fiber breakages could be seen at the interface on the fracture surface of both groups (Figs 6, 7).

EDS image analysis of the FRC substrate showed that the area percentage of polymer matrix was 32.7 (1.9) and of E-glass fiber 67.3 (1.9).

Discussion

The performance of a FRC restoration depends on the framework dimensions and design (5, 6). Even though FRC FPD has been in clinical use for only a limited period of time, clinical trials have shown that adhesive and inlay FPDs made of FRC can perform well (1–3); commonly reported failures have been delamination or chipping of the unsupported veneering composite (16). This study was therefore an attempt to investigate the reliability of several intra-oral repair methods.

One of the commonly used methods for characterizing adhesion between materials is based on shear bond strength measurements. However, shear bond strength tests have been criticized in many investigations (17, 18). Despite the criticism about the limitations of the method regarding the mixed type of stress distribution, the shear bond strength test has remained the commonly employed standard method for evaluating adhesion of dental materials, mainly because of its overall simplicity. The shear bond test is considered an appropriate method for quantifying the adhesion and bonding of repair materials (19).

There is a significant decrease in bond strengths of new resin to aged composite in water. It has been suggested that this is due to the hydrolytic degradation and softening of the polymer structure and the formation of microcracks from water storage; water storage might thus be an adequate in vitro method for aging the composite (20). To ensure in vitro aging of the FRC substrate used in this study, a more aggressive method was used, namely boiling the FRC in water.

The novel FRC material everStick, consisting of a mixture of cross-linked dimethacrylate and a linear PMMA matrix, allowed interdiffusion bonding of the new resin (6). However, to facilitate interdiffusion bonding to the polymerized framework, further wetting of the bonding site with an adhesive resin and keeping in a dark place for a minimum of 5 min were recommended (6). This allowed monomers with the same dissolving parameter as PMMA to diffuse into the polymer matrix. However, when FRC FPD is repaired intra-orally, the period of time needed for the diffusion of resin monomers to polymer might be too long for practical reasons.

Previous studies have shown that adhesive resins containing either bisGMA-hydroxyethylmethacrylate (HEMA) or bisGMA-triethylene glycol dimethacrylate (TEGDMA) improve the repair bond strength. This was explained by the improved wetting of the substrate surface by the resin and the possibility of swelling the substrate surface to some extent (9). However, the shear bond strength values obtained in the present study were lower than has been reported previously (9–11). This was probably due to the difference in the fraction of glass fibers in the polymer matrix. When the surface of FRC was ground and the glass fibers were exposed, the everStick FRC contained less polymer matrix than the previously tested Stick FRC (9). In addition, the short application time of the adhesive resin to the substrate surface might explain the lower bond strength values.

The addition of the so-called chemical adhesion primer CA, in combination with use of a resin, did not show any benefit. CA consisted of hydrophobic methylmethacrylates, which could swell the PMMA-based polymer matrix of FRC and increase the bond strength. However, this resulted in low bond strength values and two samples in the CA group failed even during thermocycling.

EDS analysis of the substrate surface in the present study revealed that the area percentage of glass fibers was 67.3% of the whole surface. Because the major proportion was glass, the use of silanes was expected to be a viable option for adhering new resin to the FRC. Application of the silane coupling agent 3-methacryloxypropyl trimethoxysilane (MPTS), in combination with the resin, clearly increased the bond strength values from 10 MPa to 23.8 MPa. This might be due to the increased wettability of the glass-rich surface, as well as to the additional benefit of the chemical bonding of silane to the OH groups of glass surface (21). Moreover, when the methacrylate groups of the silane were co-polymerized with the repair composite, the high repair strength was obvious. However, the CF group, with the same silane in combination with an adhesive primer and resin application, resulted in an even higher bond strength (35.6 MPa). This might have been due to the synergism between silane and resin based on phosphate groups (21, 22).

The results of this study support the findings of Swift (7) and Rosentritt (8), whose pretreatment of composite substrate with air-abrading particles produced satisfactory bond strength values for a new composite resin. The CJ air-abraded surface, in combination with the silane treatment and adhesive resin application, showed the highest bond strength values. Obviously, this was an additional benefit of the mechanical interlocking caused by the rough surface created after air-abrading. However, interestingly, the difference in bond strength values was not statistically significant compared to the CF group in which CF was applied to the relatively smooth substrate surface.

Thermocycling increased the bond strength values modestly in the groups with the silane treatment, whereas there was no effect on or even a decrease in bond strength values for CA and MP. This difference in behavior could have been due to the further condensation reaction of the silane-based siloxane network between the glass fiber and the resin, promoted by the increased temperature of the thermocycling process. Moreover, the heat effects of thermocycling can cause post-curing of the composite resin and stress relaxation, which could have increased the bond strength (23). On the other hand, the only group that showed a significant increase after thermocycling was the air-abraded CJ-SIL-MP group. This increase might be the result of the increased mechanical interlocking due to the previously reported (24) high water uptake of the hydrophilic resin containing HEMA at the interface. However, this hypothesis needs further investigation.

Visual examination of the fracture surfaces showed mostly cohesion type failure for all the groups with silane pretreatment, whereas an adhesion type failure was observed for the MP or CA group.

SEM examination of the surfaces confirmed the visual examination results. SEM-EDS evaluation of the air-particle abraded surface showed micro-irregularities on the glass fiber surface as well as on the polymer matrix (Fig. 5b), and a small number of particles remaining on the surface. The effect of particles remaining on the surface and of micro-irregularities created on the fiber surface is not known, and their long-term contribution to bond strength was beyond the scope of this study.

The shear bond strength data were analyzed using the Weibull analysis, in which the failure probability could be predicted at any stress level (14, 15). The quality of an estimate of failure probability depends on the number of test specimens. It was suggested that 12 test specimens might be enough (13, 25). The correlation coefficient r showed values over 0.9, except for one group showing 2 different types of behavior, thus confirming that the number of specimens ($n = 12$) used in this study was acceptable for evaluation of the data (14, 25).

It has been stated that a high Weibull modulus (= also called shape factor) (m) is desirable for all materials, since it indicates more predictable failure behavior and a more homogeneous substrate/adherend interface. Conversely, a low Weibull modulus is indicative of a large spread within the group and less predictable failure behavior. It has been suggested that generating the cumulative failure distribution of bonded materials allows for the failure probability to be estimated at different levels of stress (14, 15, 25). With this statistical approach, a more relevant clinical approximation for the risk level of failure can be made, since the low stress levels at which the first 10–20% of specimens failed were more relevant to the reliability of the bond than average strength values or very high values (26). Shear bond strength values of specimens stored in water showed similar behavior for the CF and CJ-SIL-MP groups, as seen from the cumulative Weibull distribution (Fig. 3). Although the SIL-MP silanated group showed characteristic strength of 11.7 MPa, which is clearly higher than that of CA and MP, the Weibull modulus was 1.61. The 10% failure probability was 5.5 MPa, showing practically the same stress level as the groups without silane treatment (CA (4.6 MPa), MP (5.0 MPa)). In groups CF, SIL-MP, and CJ-SIL-MP containing a silane coupling agent, both σ_0 and m values tended to increase after thermocycling, and the reliability of the bond was increased.

The results of this study suggest that bonding methods in which the use of a silane coupling agent is included, in combination with resin and air-particle abrading of the surface, are more reliable for the repair procedures of high strength multiphase FRC. The quality of the repair bond, as reflected by the low Weibull modulus, showed that use of a resin alone did not provide reliable bonding without being combined with silane coupling agent and air-abrading.

Acknowledgements.—Our study was funded through the Finnish Technology Agency (TEKES) and an IADR-CED Visiting Researcher Grant. The FRC Research Group of Turku University belongs to the Center of Excellence (Bio and Nanopolymers Research group) of the Academy of Finland, whose support is greatly appreciated. We thank Kuraray Co, 3M-ESPE, for generously supplying the materials used in the study.

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Received for publication 14 August 2003
Accepted 10 November 2003