Influence of Monomer Systems on the Bond Strength Between Resin Composites and Polymerized Fiber-Reinforced Composite upon Aging

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Purpose: This study examined the influence of different monomer systems on the tensile bond strength between a resin composite and a polymerized fiber-reinforced composite (FRC). The influence of the age (shelf-life) of the FRC prepreg (reinforcing fiber pre-impregnated with a resin system) before preparing the FRC substrate for the bonding test was also assessed.

Materials and Methods: Semi-interpenetrating polymer network (semi-IPN)-based glass FRC prepregs were aged for various durations (1, 1.5, and 3 years) at 4°C before being used to prepare FRC substrates via light polymerization. Four groups of aged prepregs were prepared through different treatments with: 1. no primer; 2. a dimethacrylate-based adhesive primer; 3. a universal primer; and 4. a specific composite primer. Subsequently, a resin composite luting cement was applied on the treated FRC substrates and cured with light. The water sorption of the FRC-composite specimens was determined. Then, the differences in the tensile bond strength were evaluated using ANOVA ($p \leq 0.05$).

Results: There were significant differences in the tensile bond strength between the composite cement and the FRC according to the primer used ($p < 0.001$), aging time ($p < 0.001$), and their interactive effect ($p < 0.001$).

Conclusion: The monomers of the universal primer demonstrated the best ability to diffuse into the semi-IPN structure of the polymer matrix of FRC. This improved the interfacial bond strength between the composite cement and the FRC substrate.

Keywords: adhesive interface, fiber-reinforced composite, poly(methyl methacrylate), primer, semi-interpenetrating polymer network, tensile bond strength.

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The past 10 years have witnessed the continuous development and use of fiber-reinforced composites (FRCs) in a wide range of clinical applications, such as endodontic posts, cores, periodontal splints, onlays, orthodontic appliances, and fixed dental prostheses (FDP).[20] The use of minimally invasive FRC for crowns and bridges is preferred to that of conventional FDP, owing to cost and time considerations. Additionally, the enhanced mechanical properties of FRC facilitate its use in fabricating frameworks of short- and medium-span FDPs.[5,10,22]

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Table 1 Materials used in the fabrication of the samples

Brand name	Manufacturer	Composition	Lot No.
everStick C&B	StickTech; Turku, Finland	Bisphenol A-glycidyl methacrylate, poly(methyl methacrylate), substituted methacrylate (<0.5%), hydroquinone (<0.5%) photoinitiator system	1612081 1606291 1412081
StickResin	GC	(1-methylethylidene)bis[4,1-phenyleneoxy(2-hydroxy-3,1-propanediyl)] bismethacrylate (25-50%), 2,2'-ethylenedioxydiethyl dimethacrylate (25-50%), 2-dimethylaminoethyl methacrylate (0.1-0.5%), photoinitiator system	5411810
Composite Primer	GC	2-hydroxyethyl methacrylate (30-60%), tetrahydrofurfuryl methacrylate (10-30%), urethane dimethacrylate (10-30%), photoinitiator system	1704031
G-Multi Primer	GC	Ethyl alcohol (90-100%), phosphoric acid ester monomer (1-5%), dimethacrylate component (1-5%)	1602041
G-Cem LinkAce	GC	Urethane dimethacrylate (25-50%), dimethacrylate (5-10%), phosphoric acid ester monomer (1-5%), dual-curing initiator system	1702024

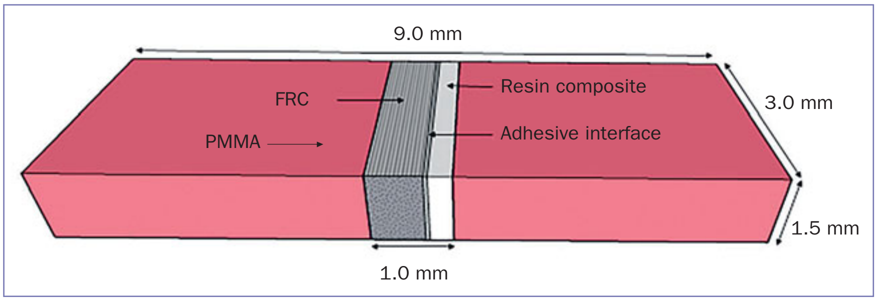

Fig 1 Dimensions of tensile test specimen.

One of the early problems with FRC restorations was weak bonding between resin and FRC.[11] With direct restorations, the adhesion of resin composite to the FRC substrate is based on free-radical polymerization. However, with indirect restorations, such as the repair of veneering composite, post, and core, the bonding mechanism with the FRC substrate is based on either micromechanical interlocking or the silanization of exposed glass fibers. However, the latter is ineffective over the long term owing to the hydrolysis of the bonds mediated by the silane coupling agent.[8]

Multi-phase FRCs, also called semi-interpenetrating polymer networks (semi-IPNs), were employed to overcome the problems associated with indirect restorations. These multi-phasic FRC materials have a unique combination of properties achieved with precise volumes of cross-linked bisphenol-A-glycidyl dimethacrylate (bis-GMA) or triethylene glycol dimethacrylate (TEG-DMA) and poly(methyl methacrylate) (PMMA).[19] The advantage of semi-IPN-based FRC is the interdiffusion of resin-composite monomer and the linear polymeric phase of the FRC substrate,[16,21] owing to the presence of an enriched PMMA layer on the FRC surface. The diffused monomer becomes interlocked after resin polymerization, resulting in strong bonding at the interface.[17]

Recently, the present authors have reported that the semi-IPN structure and the enrichment of PMMA on the surface change during the shelf-life of the FRC prepreg (a reinforcing fiber pre-impregnated with a resin system).[11] In another study, we also observed that primers with certain compositions can infiltrate into the FRC substrate.[12] Based on these studies, the present study compared the influence of different primer compositions on the tensile bond strength between FRC and resin, as well as the effect of the monomer systems on an FRC made of prepregs aged for different intervals before use. It was hypothesized that the monomer systems of adhesive primers and resin composite would affect the tensile bond strength between the resin composite and the FRC.

MATERIALS AND METHODS

Semi-IPN-based FRC prepreg specimens (everStick C&B, Stick Tech, GC Group; Turku, Finland) were refrigerated at 4 ± 1°C for three different durations: 1, 1.5, and 3 years. A silicone mold was used to fabricate FRC specimens with the uniform dimensions of 3.0 mm × 4.5 mm × 0.7 mm (width × height × thickness). The FRC prepreg was cut off

Table 2 Mean water sorption values for the tested groups (n = 6)

	Water sorption (µg/µl)			
Aging period	No primer	StickResin	G-Multi Primer	Composite Primer
1 year	19.2 ± 9.3	23.0 ± 4.6	19.7 ± 9.9	23.1 ± 4.4
1.5 years	23.6 ± 7.2	21.1 ± 12.0	22.1 ± 11.2	17.5 ± 6.2
3 years	21.9 ± 8.3	25.7 ± 4.3	23.3 ± 10.1	22.7 ± 7.5

No statistically significant differences were observed between the groups according to storage or priming condition.

Table 3 Mean and standard deviations for the tensile bond strength for the tested groups

	Tensile bond strength (MPa)			
Aging period	No primer	StickResin	G-Multi Primer	Composite Primer
1 year	$18.4 \pm 1.6^{\mathrm{Aa,b}}$	$21.1 \pm 3.7^{\mathrm{Bd,e}}$	$28.0 \pm 2.9^{\mathrm{A,Bf,g}}$	$23.1 \pm 5.6^{\mathrm{h}}$
1.5 years	$12.4 \pm 1.3^{\mathrm{C,Da,c}}$	$14.0 \pm 2.0^{\mathrm{E,Fd}}$	$17.5 \pm 2.4^{\mathrm{C,Ef}}$	$17.9 \pm 2.8^{\mathrm{D,Fi}}$
3 years	$9.1 \pm 1.1^{\mathrm{G,Hb,c}}$	$13.9 \pm 1.0^{\mathrm{Ge}}$	$15.5 \pm 2.7^{\mathrm{H,Ig}}$	$10.2 \pm 2.3^{\mathrm{Ih,i}}$

Same superscript uppercase letters demonstrate statistically significant differences between the experimental groups in different columns ($p < 0.05$). Same superscript lowercase letters demonstrate statistically significant differences between the aging groups in different rows ($p < 0.05$).

from the fiber frame, placed inside the mold, and then hand-pressed between two glass plates to obtain a smooth, even surface. Subsequently, the pressed specimens were light cured for 40 s with an irradiance of 1150 mW/cm^2 (Elipar S10, 3M Oral Care; St Paul, MN, USA). Subsequently, a single coat of different adhesive primers was applied using a fine microbrush on the FRC substrate groups with different aging times: control (no primer), bis-GMA/TEGDMA unfilled adhesive primer (StickResin, GC; Tokyo, Japan), multi-purpose primer (G-Multi Primer, GC), and composite primer (Composite Primer, GC). This yielded a total of 12 subgroups (n = 6) with different FRC aging time/primer combinations as used in a previous study.[11] The details and compositions of the materials are given in Table 1.

Subsequently, the specimens were placed under a light-protection shield (Viva Pad, Ivoclar Vivadent; Schaan, Liechtenstein) for 3 min to allow the monomer system to diffuse into the FRC substrate without the influence of ambient light. Subsequently, the specimens were light cured for 20 s. Finally, a 3-mm-thick coat of resin composite (G-CEM LinkAce, GC) was applied on the treated FRC substrates. A Mylar sheet and a glass plate were used to extrude the excess material and achieve a smooth surface. Finally, after performing light polymerization for 40 s, the specimens were polished and finished with 1200-grit silicon carbide paper under running water.

Each prepared specimen was embedded in the middle of a silicone mold (3.0 mm × 4.5 mm × 9.0 mm), and a self-curing acrylic resin (Eco-Cryl Cold, Protechno; Vilamalla Girona, Spain) was filled into both sides of the specimen (Fig 1). Subsequently, the specimen was removed from the mold and stored in a desiccator for 24 h before further processing. For tensile bond testing, the specimen blocks were sectioned with a precision diamond saw (IsoMet 5000, Buehler; Lake Blu, IL, USA) at 1400 rpm under water cooling to the dimensions of 1.5 mm × 3.0 mm × 9 mm. The edges of each sectioned specimen were finished with 1200-grit silicon carbide paper under running water.

Tensile Bond Strength Test

The tensile bond strength at the resin composite-FRC interface, the location at which the adhesive interface layer was formed, was measured using a universal testing machine (Model no. 3369 Instron; Canton, MA, USA) under tension. The specimen surface area was measured using a digital caliper (Mitutoyo; Tokyo, Japan). Each thin, rectangular plate specimen was fixed to the grips of a tensile device with cyanoacrylate glue (Super Glue, Henkel/Loctite; Westlake, OH, USA). The proprietary software of the testing machine recorded the failure loads in N and the bond strengths in MPa. A load cell of 5 kN and a crosshead speed of 0.5 mm/min were used until fracture occurred.

Sorption Test

A water sorption test was performed to stabilize the water content in the specimens for bond strength testing. Thin, rectangular plate specimens from each group were initially weighed (M_i). During the water sorption test, each specimen was immersed in a glass vial containing 50 ml of distilled water, and weighed at 1, 3, 7, and 14 days until the

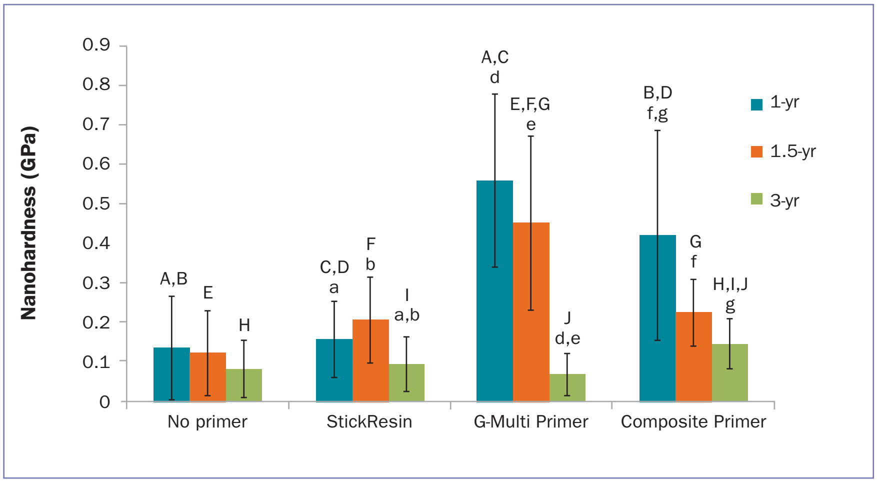

Fig 2 Nanohardness data of the adhesive interface layer using different primers. Reprinted from "Influence of primers on the properties of the adhesive interface between resin composite luting cement and fiber-reinforced composite" by Khan et al. 2018.[12]

Table 4 Linear regression coefficient table between nanohardness and tensile bond strength

	Unstandardized coefficients	Standardized coefficients		
Model	Std. Error	Beta	t	Sig.
Constant	0.93		14.27	0.000
GPa	3.13	0.514	5.02	0.000

Table 5 Pearson correlation matrix between nanohardness and tensile strength

		GPa	MPa
GPa	Pearson correlation Sig. (2-tailed) N		0.514* < 0.001 72
MPa	Pearson correlation Sig. (2-tailed) N	0.514* < 0.001 72	

*Correlation is significant at the 0.01 level (2-tailed).

weight became stable (M_f). The water sorption values (W_{sp}, μg/mm^3) were calculated with the formula $W_{sp} = (M_f - M_i)/V$, using an analytical scale (Precisa, EP 320A; Dietikon, Switzerland) accurate to 0.1 mg.

Fractographic Analysis

Fractured plate specimens were observed under a light microscope (Nikon SM2-10; Tokyo, Japan) at a magnification of 20X. The fracture type was defined by the location of the fracture: within the FRC (cohesive), at the interface between resin composite and FRC (adhesive), and with both resin composite and FRC existing in the same fragment (mixed).[13,14] The frequencies of different fracture modes were recorded for each group.

Statistical Analysis

The acquired data were analyzed using SPSS v 23.0 software (SPSS; Chicago, IL, USA). Both descriptive (means and standard deviations) and inferential statistics (two-way ANOVA) were used. Subsequently, Tukey's post-hoc test was performed to distinguish the mean differences between the variables. Furthermore, linear regression analysis was performed between the nanohardness data of our previous work[12] and the tensile bond strength data of the present study, and the bivariate (Pearson) correlation coefficients were calculated. The significance level for all statistical tests was set at $p \leq 0.05$.

RESULTS

The mean water sorption values are presented in Table 2. The water sorption values among the different groups varied from 19.2 ± 9.3 to 25.7 ± 4.3 μg/mm^3. Table 3 presents the descriptive and inferential statistics for tensile bond strengths. Two-way ANOVA revealed that both the aging time and the type of adhesive primer used had significant effects on the tensile bond strength between resin composite and FRC ($p < 0.001$). The interaction of aging time and adhesive primer used was also observed to be significant ($p < 0.01$). The post-hoc Tukey's test revealed significant differences between no primer and G-Multi Primer and between StickResin and G-Multi Primer in the groups aged for 1 and 1.5 years. At the end of 3 years of aging, G-Multi Primer was observed to have a statistically higher tensile

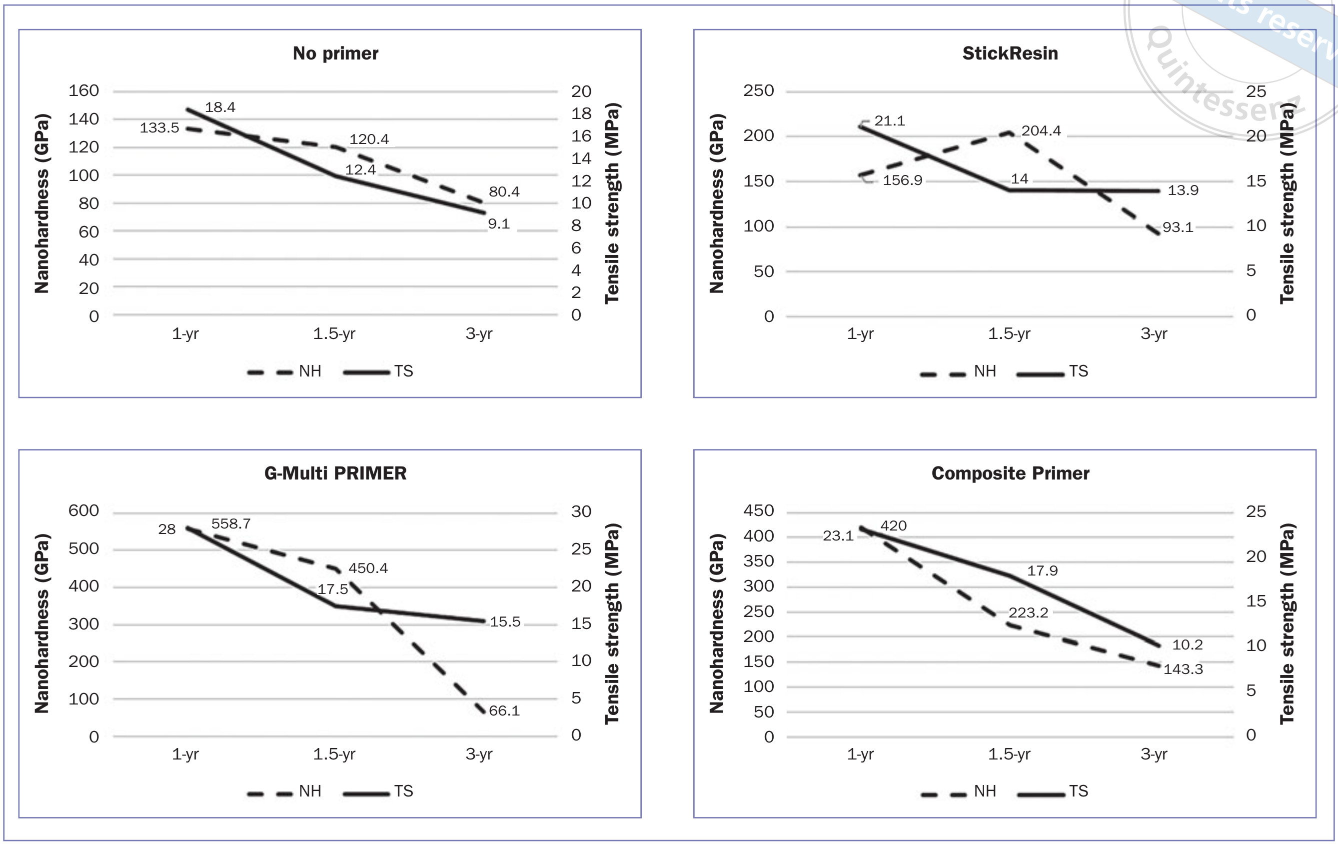

Fig 3 Correlation between nanohardness (NH) and tensile strength (TS) using various priming conditions and aging times.

bond strength (15.5 ± 2.7 MPa) compared with no primer (9.1 ± 1.2 MPa) or Composite Primer (10.2 ± 2.3 MPa).

The regression analysis results in Table 4 suggest a positive linear relationship between the nanohardness and tensile bond strength ($p < 0.001$). A corrected R^2 value of 0.265 was observed with the statistical difference at $p \leq 0.05$. The Pearson correlation coefficient value was determined to be 0.51, suggesting a strong correlation between the nanohardness and tensile bond strength (Table 5). The correlations are presented graphically in Fig 3.

Most of the failures were adhesive (Table 6). Adhesive failures (AD) were dominant for the FRCs with no primer, irrespective of the aging time. However, mixed and cohesive failures (MI and CO, respectively) were observed when using the G-Multi Primer and Composite Primer. At the end of 3-year aging, G-Multi Primer showed 16.7% mixed failure, whereas all other groups demonstrated 100% adhesive failure.

DISCUSSION

Our original hypothesis was contradicted by the findings of this study, because the monomer systems of the primers used here showed a significant influence on the tensile bond strength between the resin composite and the FRC substrate. The study was designed to examine the nature of the adhesive interface between the resin composite and a dental FRC substructure using different monomer systems. As the bond strength is a useful clinical predictor for the successful adhesion of dental restorations to tooth substances,[7,9] this parameter was chosen to evaluate the bonding properties of resin composite to FRC at different aging times during the shelf-life of the FRC.

Dental composites absorb water to different degrees depending on the polarity of the molecular structure, the presence of hydroxyl groups, and the degree of crosslinking in the continuous matrix. Hence, we considered water sorption a key property that influences the bond strength.[15] However, our results did not show significant differences in water sorption between various adhesive primers and resin composite.

The tensile bond strength data suggest that the application of G-Multi Primer on the FRC substrate enhanced the bond strength of resin composite to FRC. This might be due to the presence of a relatively large amount of ethanol in G-Multi Primer, which swelled the linear polymer of PMMA in the semi-IPN system of the FRC; consequently, the phosphate ester monomer and dimethacrylate component could penetrate into the semi-IPN structure for enhanced bonding. The presence of ethanol is known to increase the polymer

Table 6 Failure mode distribution among the study groups

	Storage duration								
	1 year			1.5 years			3 years		
	AD	MI	CO	AD	MI	CO	AD	MI	CO
Groups		%			%			%	
No primer	83.3	16.7	0	100	0	0	100	0	0
StickResin	66.7	16.7	16.7	100	0	0	100	100	0
G-Multi Primer	50	33.3	16.7	66.7	16.7	16.7	66.7	16.7	16.7
Composite Primer	50	33.3	16.7	83.3	16.7	0	100	0	0

AD = adhesive failure, MI = mixed failure, CO = cohesive failure.

chain mobility and radical diffusion rate.[3] The effect of ethanol on PMMA has been studied in more detail elsewhere.[1,2]

The increased tensile bond strength in the group using Composite Primer might suggest that the monomers in this system (2-hydroxyethyl methacrylate [HEMA, 30% to 60%] and urethane dimethacrylate [UDMA, 10% to 30%]) dissolved the FRC surface and improved the penetration of resin composite monomers (G-Cem LinkAce). The reason could be the lower molecular weight (MW) and viscosity (η) of the monomers in Composite Primer: MW = 130.14 g/mol, η = 0.0057 Pa for HEMA; MW = 470 g/mol, η = 23 Pa for UDMA.[6] However, during its shelf-life, the prepreg might gradually lose the crosslinking monomers on its surface and become enriched with PMMA molecules. Hence, lower tensile bond strengths were observed.

The data for StickResin suggest that the monomers of the resin composite could not properly dissolve the FRC surface in this case. The base monomer in StickResin, ie, bis-GMA, has a molecular weight of 512 g/mol, which is higher than that of HEMA and UDMA. Furthermore, despite the high intrinsic reactivity of bis-GMA, hydroxyl groups on the backbone and the interactions enabled by the aromatic rings increase the initial viscosity to η = 1200 Pa. The viscous primer resin reduced monomer mobility during polymerization,[3,4] and hence, the primary goal of dissolving the FRC surface could not be achieved using this monomer system. Moreover, although the solubility parameters of bis-GMA are close to those of PMMA (the linear polymeric component of this particular FRC), it is possible that the photoinitiator system in StickResin hindered the dissolution ability of bis-GMA and therefore lowered the tensile bond strength of the adhesive interface for this group.[12,17] Our previous work showed a slightly higher degree of monomer conversion (DC%) at the interface between FRC and resin composite when the resin composite was used with StickResin primer compared with the use of other primers.[12] The presence of 25% to 50% UDMA in G-Cem LinkAce resin composite suggests that UDMA alone could not sufficiently swell the surface of the FRC substrate owing to its high molecular weight.

In all the groups, the tensile bond strength decreased with the increase in the aging time of the FRC prepregs. This suggests that the polymeric structure in the prepregs might gradually change with time, resulting in the enrichment of the linear polymer PMMA at the interface. This change could be due to the diffusion of the crosslinking monomers and the dissolved PMMA molecules in the FRC prepreg. Consequently, the overall tensile bond strength of resin composite and FRC was lower for the prepregs aged for 3 years among all the primer groups.

The bonding layer thickness might have affected the tensile bond strength of the material system. When the monomers of the resin composite diffused into the IPN layer of the FRC substrate, the interdiffusion zone had a lower crosslinking density than the cured resin composite of the dimethacrylate system. Thus, from the polymeric structural point of view, the cured resin composite layer has a higher tensile bond strength than the interdiffusion zone between the FRC and the resin composite. Although the limiting factor for the strength of the interface system was the interdiffusion zone, the stress distribution between the polymer layers of various thicknesses and different physical properties was assumed to influence the measured tensile bond strengths. Furthermore, a slight variation in specimen geometries might have affected the tensile bond strength. However, a previous study suggested that, for a given cross-sectional area and for any gauge length, variation in the specimen geometries has no effect on the tensile bond strength.[18]

The variability of tensile bond strength can be explained by the nanohardness of the cured FRC material, as a clear correlation between the two was generally observed. A similar declining trend in the nanohardness values was also observed with the aging of the prepregs before their polymerization in our previous study.[12] The lower R^2 value might be due to the use of StickResin, which showed increasing nanohardness at 1.5 years, whereas the tensile bond strength was lower at 1 year than at 1.5 years.

It was observed that the failure mode tends to shift from adhesive to cohesive with increased interfacial bond strength

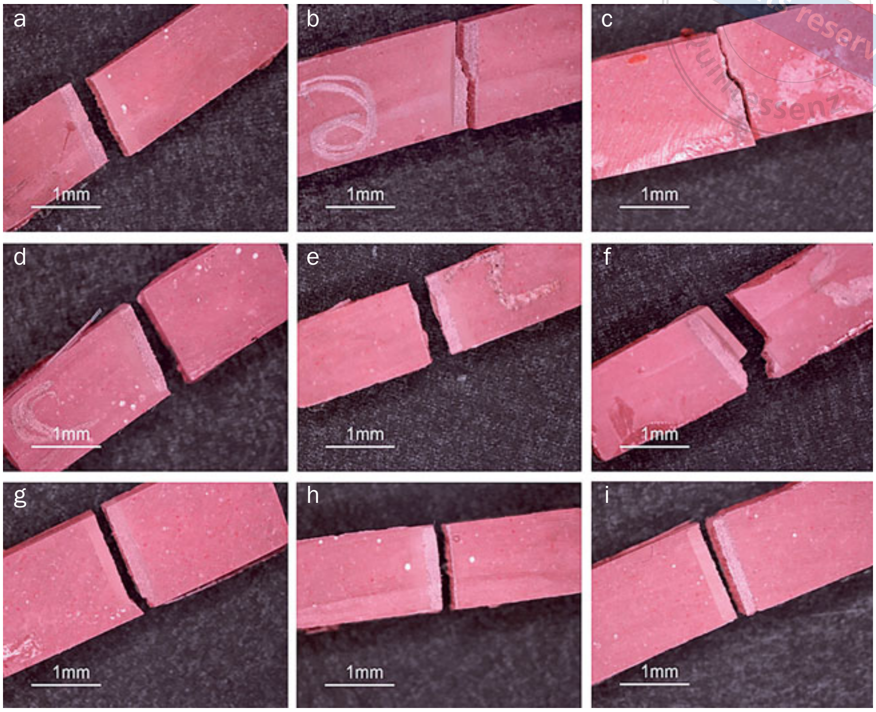

Fig 4 Stereomicroscopic images of specimens using prepregs aged for 1 year (a–c). a: adhesive failure with no primer; b: mixed failure with G-Multi Primer; c: mixed failure with Composite Primer. Images of specimens using prepregs aged for 1.5 years (d–f). d: adhesive failure with StickResin; e: cohesive failure with G-Multi Primer; f: mixed failure with Composite Primer. Images of specimens using prepregs aged for 3 years (g–i). g: adhesive failure with StickResin; h: cohesive failure with G-Multi Primer; i: adhesive failure with Composite Primer.

between the resin composite and the FRC. Both G-Multi Primer and Composite Primer showed more mixed failures (33.33% and 16.66% after aging for 1 and 1.5 years, respectively) compared with the other groups. StickResin, G-Multi Primer, and Composite Primer all exhibited 16.66% cohesive failures with the prepreg aged for 1 year, indicating that the adhesion between resin composite and FRC was stronger than the cohesive strength of the FRC substrate. However, cohesive failure for the prepregs with G-Multi Primer aged for 1.5 (Fig 4e) and 3 years (Fig 4h) suggests that the FRC components of the IPN system might disintegrate during prolonged storage, resulting in the dual phases or decreased cohesive strength of the matrices.

The findings of this study corroborate the durability of the clinical repair bond strength of FRCs. Although the current tensile bond strength data suggest a reduction in the bond strength with the aging of the prepregs, good repair bond strength is provided even after 3 years of clinical service.

While this study only included commercially available primers composed of multiple monomers and other solvents, future studies of individual monomers should be conducted to explain the results. In addition, only one type of resin composite was used in this study, and the present results should be verified with other resin composites. Moreover, prospective clinical studies are required to evaluate whether the improved bonding in G-Multi Primer observed in this study corresponds to enhanced clinical performance.

CONCLUSIONS

It is likely that the shelf-life of the FRC prepreg and the priming monomer systems both influence the bond strength between resin composite and FRC substrate. Both universal and composite primers show enhanced tensile bond strength with prepregs aged for 1 and 1.5 years. Only GC Universal Primer showed higher tensile bond strength with prepregs aged for 3 years. The lower bond strength with older prepregs for all primer conditions might suggest a diffusion-related change of the semi-IPN polymer matrix before the prepreg was used.

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Clinical relevance: The findings of this study corroborate the durability of the clinical repair bond strength of semi-IPN based FRC. The use of such priming monomer systems with specific compositions may efficiently dissolve the enriched PMMA layer on the semi-IPN based FRC substrate, thus promoting sufficient repair bond strength between a resin composite and aged FRC even after 3 years of clinical service.