Measuring the effects of water immersion conditions on the durability of fiber-reinforced hybrid composite resin using static and dynamic tests

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The present study was designed to measure the durability of glass fiber-reinforced hybrid resins (FRC) in clinical applications. Accordingly, we studied the effects of static and dynamic loading as well as temperature changes inside the oral cavity, a moist environment, on the bending strength of FRC. The bending strength was measured using several tests, including an open-air bending strength test (AE), a 24-h water immersion test (WC1D), a 2-year water immersion test (WC2Y), a thermal cycling test (TC), a repeated in-water impact test at 37°C/10⁵ (WI37), and a repeated in-water impact test at 55°C/10⁵ (WI55). The following tests are ordered from greatest to least with respect to GF's bending strength: AE, WI37, WI55, WC1D, WC2Y, and TC. Likewise, the following tests are ordered from greatest to least with respect to EV's bending strength: AE, WC1D, WC2Y, WI37, WI55, and TC.

Keywords: Glass fiber, Hybrid resin, Static and dynamic test

INTRODUCTION

Minimal intervention is important in planning enamel and/or dentin restoration in order to leave the healthy part of the tooth as intact as possible^{1,2)}. When fixed partial dentures are used to restore missing teeth, the healthy parts of the tooth are drilled off to obtain sufficient mechanical strength and retention and prevent detachment even if both adjacent teeth are intact.

The application of adhesive dentistry for abutment teeth has enabled the use of glass fiber-reinforced (FRC) fixed partial dentures. These allow minimally invasive tooth drilling and address aesthetic demands and health issues, including metal allergies. The mechanical strength of fixed partial dentures made of composite materials is structurally reinforced by glass fibers, and their clinical application has been previously studied^{3–21)}.

The bonding property at the reinforcement/matrix interface is a critical factor affecting the mechanical properties of composite materials. In particular, the addition of filler particles to a bonding agent and substantially active silane-coupling treatment enhance interfacial bonding between the fibers and the hybrid resin matrix, improving the flexural strength of FRC^{21–24)}.

In clinical practice, patients receiving prosthetics made of such materials have numerous concerns, including safety, durability, breakage, the effect of brushing on the shiny appearance, and whether to expect marginal leakage. Further studies on these issues should be translated into how cyclic impact loads by occlusion and mastication as well as the aqueous environment of the oral cavity affect deterioration of the hybrid resin and the glass fiber framework. This information will help provide guidelines for clinical application.

In this study, the effects of temperature changes in the moist oral environment and cyclic impact loads on

the flexural strength of plate specimens were investigated to describe the material durability of FRC for clinical applications. In addition, fractured surfaces were analyzed by scanning electronic microscopy.

MATERIALS AND METHODS

Materials

Hybrid resin ES (Estenia, Kuraray Medical, Tokyo, Japan) and glass fibers GF and EV (Estenia C&B EG Fiber, Kuraray Medical, Tokyo, Japan, and everStick C&B, StickTech, Turku, Finland) were used in this study as the resin and reinforcement materials, respectively. The compositions of the materials used in this study are listed in Table 1. Figure 1 shows the glass fibers and a fiber-reinforced specimen as well as a view of a frame of specimen.

Methods

In this study, 3-point bending FRC specimens were subjected to either static loading or dynamic loading in order to investigate its flexural strength. Static loading conditions included an air environment at 23°C for 24 h (AE), water conditions at 37°C for 24 h (WC1D), water conditions at 37°C for 2 years (WC2Y), and thermal cycles of 5°C to 55°C (TC). Dynamic loading conditions included a water impact load of 37°C/10⁵ (WI37) and a water impact load of 55°C/10⁵ (WI55).

This was a 2-way experimental study, with factors A and B. Factor A was the type of fiber (GF and EV), and factor B was the experimental condition (AE, WC1D, WC2Y, TC, WI37, WI55). Thus, there were 12 combinations, which were repeated 6 times for a total of 72 randomized experiments.

Table 1 Code, Manufacturer, Lot No., Principal ingredient, light, and heat polymerization time of materials used in this study

Code	Manufacturer	Lot No.	Filler content (%)	Monomer	Filler	Polymerization
Hybrid Resin						
ES	Kuraray Medical, Tokyo, Japan	0038C	92	UTMA	SiO ₂ , BaO ₂ , Al ₂ O ₃ , La ₂ O ₃	Polymerization for 90 s Light curing, 180 s Heat curing 110°C, 15 min
Reinforced Fiber						
GF	Kuraray Medical, Tokyo, Japan	0023A	30	UTMA, UDMA, TEGDMA	Microfiller surface treatment	
EV	Ever Stick C&B, Turck, Finland	2070917-ES-19	0	Bis-GMA, PMMA

Fig. 1 Glass fibers (a and b) and glass fiber-reinforced hybrid resin test piece (c) used in this study.

Specimen preparation

Plate-like specimens measuring 3.0×4.0×40.0 mm were prepared according to the ISO specifications (ISO 14704)²⁵⁾. The specimens prepared with ES and with GF/EV in the present study weighed 1.18 g and 0.191 g, respectively. Consequently, the weight percentage of GF/EV was 16.2%. GF/EV was cut to a length of 40.0 mm, placed in the direction of its long axis, and pressed flat against the base of a stainless steel split mold (Fig. 2). Then, ES was filled over the GF/EV, and both sides of the mold were covered with glass plates, which were gently pressed and fixed in place with clips. The thicknesses of GF/EV and ES placed on the specimen (3 mm) were

approximately 1.0 mm and 2.0 mm, respectively. The specimens were light-polymerized with a light-curing device (Dentacolor XS, Kulzer, Wehrheim, Germany) from both sides of the mold for 90 s for a total of 180 s. They were subsequently transferred into a heat-curing oven (KL-100, Kuraray Medical, Tokyo, Japan) for heat activation at 110°C for 15 min. Subsequently, the edges of the specimens were polished with #600 waterproof abrasive paper and stored in a temperature-controlled water bath at 37°C for 24 h before testing. The preparation of each specimen was completed under temperature and humidity conditions of 23°C±2°C and 50%±5%, respectively.

Fig. 2 View of a frame of a stainless steel split mold.

Fig. 3 View of a frame of the flexural strength test.

Flexural strength test

The specimens that underwent the loading conditions were mounted on a flexural strength test machine compliant with the 3-point testing method for fine ceramics, connected to an electrohydraulic material testing machine (Servopulser EHF-FD1, Shimadzu, Kyoto, Japan), and subjected to the bending test at a crosshead speed of 0.5 mm/min with a test span of 30 mm. Specimens were positioned so that GF and EV were at the tensile sides. The loads causing specimen fracture were determined in order to calculate the flexural strengths using the following equation (Fig. 3):

$$\sigma = 3PL / (2bh^2)$$

σ is the flexural strength (MPa); b is the width of the specimen (mm); P is the breaking load (N); h is the thickness of the specimen (mm), and L is the test span (mm).

Fig. 4 View of a frame of the cyclic impact loading test.

Cyclic impact loading test

The cyclic impact load testing machine (test model, Japan Mecc, Tokyo, Japan) was used with a test span of 30 mm. The frequency of cyclic impact loading and the maximum load level were set to 1 Hz and 100 N, respectively. To simulate the intraoral environment, specimens were tested while immersed in water at 37°C and 55°C (Fig. 4).

As the mean occlusal force during mastication is between 30 and 80 N, and the hand force clinically applied for seating a prosthesis is no more than 100 N. Therefore, the impact loads were set to 100 N in the present study.

Water immersion test

For water immersion, 2 conditions were chosen: 1-day and 2-year immersion in distilled water at 37°C.

Thermal cycle test

The test machine (Thermal Cycling HA-K178, Tokyo Giken, Tokyo, Japan) used a thermal cycle including: immersion for 30 s in a 5°C water bath, a 5-s transfer, and immersion for 30 s in a 55°C water bath for 5,000 successive cycles.

Fractured surfaces analysis by scanning electronic microscopy

Specimens fractured by cyclic impact loading and during the residual flexural strength test were coated with

osmium (Neoc-AN, Meiwafohis, Osaka, Japan). Observations were conducted with a scanning electron microscope (SEM, S-4000, Hitachi, Tokyo, Japan) at 20 kV. The fractured specimens were cut just below the load point, and observations were conducted primarily on the aspects of interfacial adhesion directly below the load point between the ES and the GF and EV.

Statistical analysis

Statistical processing was performed using a 2-way layout method ($\alpha < 0.05$). Twelve combinations of the 2 factors, A (the type of fiber: GF and EV) and B (the test environment: AE, WC1D, WC2Y, TC, WI37, and WI55), were tested 6 times each for a total of 72 randomized tests.

RESULTS

Flexural strength of each type of fiber and test condition

Figure 5 shows the effects of the type of glass fiber and test condition on flexural strength. Table 2 shows the results of 2-way analysis of variance. There were significant differences between factors ($p < 0.01$) and the 95% confidence interval.

The flexural strength of the GF-reinforced specimens was 745.5 MPa for AE, 652.1 MPa for WI37, 643.2 MPa for WI55, 566.3 MPa for WC1D, 273.7 MPa for WC2Y, and 160.2 MPa for TC. The rates of decrease in flexural strength of these specimens under the test conditions compared to those under an air environment were 78.5% for TC, 63.3% for WC2Y, 24.0% for WC1D, 13.7% for WI55, and 12.5% for WI37. The flexural strength of EV-reinforced specimens was 726.7 MPa for AE, 601.3 MPa for WC1D, 353.7 MPa for WC2Y, 353.4 MPa for WI37, 272.8 MPa for WI55, and 155.4 MPa for TC. The rates of decrease in flexural strength of these specimens under each test condition compared to those for the air environment were 78.6% for TC, 62.5% for WI55, 53.3% for WC2Y, 51.4% for WI37, and 17.3% for WC1D. The cyclic impact load and temperature change reduced the flexural strength of GF- and EV-reinforced specimens.

Tables 3 and 4 show the results of Tukey's multiple comparison tests. The differences between each combination of different fibers and experimental conditions were significant except for the differences between AE (GF) and AE (EV), WC1D (GF) and WC1D (EV), TC (GF) and TC (EV), WC2Y (GF) and TC (GF), WI37 (GF) and WI55 (GF), and WI37 (EV) and WI55

(EV).

Fracture morphology and SEM observation of fractured surface

Figures 6a–f show SEM images of GF and EV on the left and right, respectively, with AE on the top, WC1D in the middle, and WC2Y on the bottom. Figures 6g–l show SEM images of GF and EV on the left and right, respectively, with TC on the top, WI37 in the middle, and WI55 at the bottom. In the SEM image of the fractured AE and WC1D specimens, cohesion failure starting just below the loading point was predominantly observed for both GF and EV materials (Figs. 6a–d). Cohesion failure was also apparent in the SEM images of the fractured WC2Y specimens, and fracture lines between glass fiber and matrix resin extending to both ends or parallel to the interface were observed in the interface area for both GF and EV materials (Figs. 6e, f). In the SEM images of the fractured TC specimens, cohesion failure and mixed failure with interface fracture were observed for both GF and EV materials. In the interface area, failure due to water absorption by the matrix resin covering the glass fiber as well as at the interface between ES and matrix resin or glass fiber was also present (Figs. 6g, h). The SEM images of fractured WI37 and WI55 specimens showed dominant cohesion failure for GF without cracks and delamination in the interface area for each test condition (Figs. 6i, k). However, there was interfacial failure accompanied by delamination of ES from the

Fig. 5 Effect of different test conditions on flexural strength.

Table 2 Result of 2-way ANOVA

Source	s.s.	d.f.	m.s.	Fo
A: Reinforced materials	198002	1	198001.90	59.37**
B: Test condition	2293842	5	458768.48	137.56**
A×B	514666	5	102933.24	30.86**
Error	200104	60	3335.06	
Total	3206614	71		

** $p < 0.01$

Table 3 Tukey's multiple comparison tests of flexural strength of GF reinforced hybrid resin

	Water Impact Load 55°C/10 ⁵	Water Impact Load 37°C/10 ⁵	Thermal cycle 5–55°C/5,000	Water condition 37°C/2Y	Water condition 37°C/24 h
Air environment 23°C/24 h			**	**	**
Water condition 37°C/24 h			**	**	
Water condition 37°C/2Y	**	**	**		
Thermal cycle 5–55°C/5,000	**	**			
Water Impact Load 37°C/10 ⁵					

p*<0.05 *p*<0.01

Table 4 Tukey's multiple comparison tests of flexural strength of EV reinforced hybrid resin

	Water Impact Load 55°C/10 ⁵	Water Impact Load 37°C/10 ⁵	Thermal cycle 5–55°C/5,000	Water condition 37°C/2Y	Water condition 37°C/24 h
Air environment 23°C/24 h	**	**	**	**	**
Water condition 37°C/24 h	**	*	**	**	
Water condition 37°C/2Y			**		
Thermal cycle 5–55°C/5,000	**	**			
Water Impact Load 37°C/10 ⁵					

p*<0.05 *p*<0.01

glass fiber strained by the cyclic impact loading for ES under each test condition. Considerable amounts of residual resin were also present for GF and EV (Figs. 6j, l).

DISCUSSION

Effects of the type of glass fiber

The lowest rates of decrease were observed for WI37 (12.5%) and WC1D (17.3%) for GF and EV, respectively. GF is a fiber framework material pre-impregnated with cross-linking monomers of urethane tetramethacrylate (UTMA) and urethane dimethacrylate (UDMA) that contain 30% filler by weight. The bonding agent impregnated in EV consists mainly of bisphenol A-glycidyl methacrylate (Bis-GMA) and polymethyl methacrylate (PMMA) and contains no filler. The surface of the glass fiber is treated by silane coupling and coated with PMMA resin. In the water immersion test, similar decreases in strength were found for both GF and EV in the following order: WC1D>WC2Y>TC. In contrast, for WI37 and WI55, the strength of EV decreased by about 50% compared to that of GF, highlighting the differences

between the types of glass fiber. This may have been due to the differences in filler content of each type of fiber.

Water infiltrated into the fiber-reinforced composite material by 3 mechanisms: diffusion from the resin surface, infiltration through surface cracks and other defects, and infiltration along the interface between the fibers and the matrix. The factor leading to the greatest early-stage damage from water absorption appeared to be moisture penetrating into the surface. For static testing, the water immersion test showed an equivalent decrease for both GF and EV in the following order: WC1D>WC2Y>TC. Unlike dynamic testing, GF was less than EV. This shows that the polysiloxane interconnection network between the silanol groups of the silane treatment agent and the silanol groups of the E-glass fiber surface used in GF is easily hydrolyzed. Further, E-glass contains about 8% B₂O₃, which is easily dissolved in water. Accordingly, strength degradation is a concern in a moist environment. Matsukawa *et al.*²⁶⁾ found that the amount of water absorbed by composite resins in molar teeth increases over time for 3 years. The present study also identified long-term water absorption in the long-term water immersion test. Presumably, the

Fig. 6 a–f: SEM micrographs of fiber (GF or EV)-reinforced composites fractured by 3-point bending and static and dynamic tests.

flexural strength decreased, because starting points for cracks easily form when the FRC becomes embrittled and moisture penetrates the adhesion surface. The flexural strengths of GF and EV after the TC test were 160.5 MPa and 155.4 MPa, respectively, indicating a similar degree of decrease as previously reported by Mitobe *et al.*²¹⁾.

Effects of cyclic impact loading

The flexural strengths after 10^5 cycles of impact loading of at 37°C and 55°C differed between GF and EV, and the strength of EV decreased by about 50% compared to that of GF. This may have been attributable to the different filler contents in the bonding agents used for each glass fiber.

Fig. 6 g–l: SEM micrographs of fiber (GF or EV)-reinforced composites fractured by 3-point bending and static and dynamic tests.

Shimizu *et al.*²³⁾ also found that flexural strength varies depending on filler content. They measured flexural strength when the filler content was 0, 10, 20, 30, and 40% by weight. The flexural strength was lowest at a content of 0% and greatest at 30%. Flexural strength was reduced at a content of 40%. Since the addition of 30% of filler by weight improved durability, 30% was

considered the ideal content. These results support those of the cyclic impact load test simulating occlusal pressure, which showed the superior flexural strength of GF containing 30% filler compared to that of EV containing 0% filler.

Hashimoto *et al.*²⁷⁾ determined the bonding strength for 10 combinations of 2 types of bonding agents with

different primary monomers and 5 types of composite resins. They found the greatest strength for the combination of both containing Bis-GMA as a primary monomer. However, this was not significantly different from other combinations with different composite resins.

Compared to EV, cyclic impact loading had no major effect on the remaining bending strength of a combination of reinforcement by ES, the primary component of which is UTMA, and GF, the primary components of which are UTMA and UDMA. This may have been due to their multifunctional methacrylate, which contains the same urethane-based monomers with similar bonding characteristics.

Even with different combinations of hybrid resins and main monomers in the fiber bending material, if one considers the effects of differences in component monomers, a reduction in bending strength is insufficient to constitute a significant difference²¹⁾. It also seems that 100,000 rounds of cyclic impact loading does not lead to moisture penetration until after 14 h. Afterwards, there are critical defects in the adhesive strength. Because GF>EV, it is hypothesized that remaining flexural strength is greatly affected by different filler contents of the bonding materials in the glass fibers.

Effects of temperature change

The flexural strengths of specimens after exposure to AE, WC2Y, TC, WI37, and WI55 were determined. The decrease in flexural strength was high for specimens after applying 37°C for 2 years and 5,000 thermal cycles of 5°C to 55°C.

Säilynoja *et al.*²²⁾ suggested that temperature changes reduce flexural strength due to internal stress generated within the material. Both materials used in this study were composite materials containing matrix resin, glass-based filler, and glass fibers, which have different coefficients of thermal expansion. Thermal cycles caused material expansion and constriction, leading to continuous, complex patterns of internal stress at the interfaces. Furthermore, the increased interface stress resulted in microcracks at the interface. Additional water immersion decreased the bonding strength between ES and FRC, which was a major cause of decreased strength and led to moisture intrusion into the resin and bonding materials and hydrolysis of the silane-coupling agent.

In the oral cavity, FRC is subjected to cyclic impact loads from occlusion as well as temperature changes when exposed to saliva, food, and drink, which can significantly affect the FRC in everyday life²⁷⁻³²⁾. To eliminate these negative influences, studies on composite materials that achieve better coupling between glass fiber and resin as well as studies on optimal glass fiber designs for dental restorations are needed.

SEM analysis

As previously described, fiber-reinforced composite materials break *via* interface failure, where fractures occur preferentially at the adhesive interface, and *via* cohesive failure, where the matrix and the reinforcing

material fall apart. Interface failure occurs when there is a large difference in mechanical strength between the matrix and the reinforcing material or when the interface has inadequate adhesion. The SEM photographs from AE, WC1D, WI37 (GF), and WI55 (GF) reveal cohesive failure from directly below the loading point; interface failure was not observed. Since the remaining flexural strength was high, it appeared that cracks progressed to the interface from directly below the loading point. This indicates that the glass fiber reinforcement had a major effect. The SEM photographs from WC2Y, TC, WI37 (EC), and WI55 (EV) illustrate that interface failure and mixing failure were dominant and that breakage occurred in tandem with interfacial cracks, inter-layer peeling, and water absorption. Flexural strength also decreased along with the degree of interface failure. These results implicate cyclic impact loading and disintegration due to water absorption by the matrix resin coating the glass fibers and the ES interface between the matrix resin and the ES and the glass fibers in the interfacial failure. They also show that the glass fiber reinforcement was not effective.

CONCLUSION

This study investigated the effects of temperature changes and cyclic impact loads in the moist environment of the oral cavity on flexural strength of plate specimens in order to elucidate the durability of glass fiber-reinforced hybrid resin for clinical application. In addition, the fractured surfaces were analyzed by scanning electron microscopy. The results are summarized below:

1. The flexural strength of GF-reinforced specimens was maximal in the AE test followed by WI37, WI55, WC1D, WC2Y, and TC. The flexural strength for EV decreased in the following order: AE, WC1D, WC2Y, WI37, WI55, and TC.
2. The flexural strength of both GF and EV glass fiber-reinforced specimens decreased by 78% after exposure to TC.
3. After exposure to WI37 and WI55, the strength of EV significantly decreased by about 50% compared to GF.
4. The SEM images of fractured specimens of both GF and EV demonstrated cohesion failure and mixed failure with interface fracture.

Taken together, the above results indicate the impact of water immersion on the durability of glass fiber-reinforced hybrid resin exposed to static and dynamic loads. In clinical applications, careful attention must be paid to the status of a prosthetic device and to the materials used. Further study is needed to better understand these parameters.

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