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Fatigue Strengths of Particulate Filler Composites Reinforced with Fibers

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The aim of this study was to evaluate the dynamic fatigue strengths at 10^5 cycles and the strains of particulate filler composite resins with and without reinforcing fibers. An UHMWPE (Ribbond), a polyaromatic polyamide fiber (Fibreflex), and three glass fibers (GlasSpan, FibreKor, Vectris Frame) were used to reinforce the particulate filler composite resins. The fatigue properties were measured in three-point bending mode using a servohydraulic universal testing machine at a frequency of 5 Hz, until failure occurred or 10^5 cycles had been completed. The fatigue strengths at 10^5 cycles were determined by the staircase method. The fractured aspects of specimens were evaluated by an optical and scanning electron microscope. The fatigue strengths of particulate filler composite resins were 49-57 MPa, and those of fiber-reinforced were 90-209 MPa. Unidirectional glass fibers showed higher reinforcing effects on the fatigue strengths of composite resins. The strain of UHMWPE-reinforced composite was largest.

Key words: Fiber-reinforced composite, Dynamic fatigue strength, 3-point bending

INTRODUCTION

A composite is a multiphase material that is artificially made and separated by a distinct interface. Many composite materials are composed of two phases; one is the matrix, which is continuous and surrounds the other phase which is filler, often called the dispersed phase[1]. The dispersed phase can be particle or fiber. The reinforcing efficiency of fibers depends on the component and geometric orientation of fiber, the ratio of fiber to resin, and the adhesion between fiber and resin matrix[2−4]. Glass fiber has a high strength but its elastic modulus is not so high, while carbon fiber is relatively stiff. Polyaramid fiber is also strong and stiff[1]. The mechanical properties, particularly the impact strength of UHMWPE (ultra high molecular weight polyethylene) fiber is known to be better compared with those of glass fibers[5]. The demand for restorations using these fibers, which can replace the metal substructure, has increased due to their advantages like higher esthetic property, simple laboratory procedure, and being free of corrosion and metal allergy[6]. They can be used in periodontal splints, fixation of avulsed teeth, endodontic posts, reinforcement of denture frameworks and orthodontic appliances, and fixed partial dentures in the dental field[7].

In a previous paper, we reported on the flexural strength and elastic modulus of FRC (fiber-reinforced composite) in a static test[7]. However, the stress applied in the mouth is generally low and repeated rather than being a single impact. It is estimated that the intraoral stress received by dental restorations during mastication is repeated more than 3×10^5 times per year[8]. From this viewpoint, it might be more appropriate to estimate the strength of dental prosthesis based on the fatigue property rather than static. As in the previous paper, we made specified specimens according to ISO specification 10477[9], in order to evaluate the effect of fiber itself and to exclude the influence of design of crown and bridge on the strength and crack propagation.

Fatigue is a form of failure that occurs in structures subjected to dynamic and fluctuating stresses. Under those circumstances it is possible for failure to occur at a stress level considerably lower than the tensile or yield strength for a static load. The fatigue failure mechanism in fiber-reinforced composites is quite different from those in monolithic, homogeneous materials such as metals. The damage in FRC accelerates and then decelerates with cycling, while it accelerates monotonically in homogeneous materials[2]. The factors that affect fatigue life include mean stress level, geometrical design, surface effects, and environment[1,10]. Fatigue properties are potentially sensitive to degradation in an aqueous environment by the plasticization effect of water[11−13]. We hypothesize that dynamic fatigue results in fiber-matrix debonding and a reduction of flexural strength in a static test.

The aim of this study was to evaluate the reinforcing effects of various fibers on particulate filler composite resins by repeated loading. By obtaining the fatigue strength of FRC at 10^5 cycles, the relationship between static flexural strength and fatigue strength was investigated.

MATERIALS AND METHODS

Specimen preparation

The materials used in this study including five brands of fibers and four kinds of particulate filler composite resins are explained in Table 1. The SEM images of five fibers are shown in Figs. 1 and 2. The schematic diagram of the FRC test specimen is described in Fig. 3. A UHMWPE fiber (RB), a polyaromatic polyamide fiber (FF), and three glass fibers (FK, GS, and VF) were used. VF is a bidirectional fiber weave, while the others are continuous unidirectional fibers. One layer of each fiber

Table 1 Materials Used in the Study

Brand name	Code	Manufacturer	Lot number	Primary composition	Type (Diameter, μm)
Ribbond	RB	Ribbond Inc. WA, U.S.A.	9518	UHMWPE	Leno-woven ribbon (10-15)
GlasSpan	GS	GlasSpan Inc. PA, U.S.A.	990101	E-glass	Braided ribbon (4-6)
Fibreflex	FF	BioComp CA, U.S.A.	9801	Polyaromatic polyamide (Kevlar-49)	Unidirectional tuft (13-17)
FibreKor	FK	Jeneric/Pentron CT, U.S.A.	18529	S-glass	Unidirectional fiber Pre-impregnated in Bis-GMA (6-9)
Vectris Frame	VF	Ivoclar/Vivadent Liechtenstein	895 033	E-glass	Bi-directional woven sheet Pre-impregnated in Bis-GMA (4-6)
C&B cement	CB	Bisco IL, U.S.A.	9800001438	Bis-GMA, TEG-DMA	Self-cure
Aelitefil	AF	Bisco IL, U.S.A.	9800000839	UDMA, Bis-GMA, TEG-DMA	Light-cure
Sculpture Body	SC	Jeneric/Pentron CT, U.S.A.	801902	PCDMA, Bis-GMA	Light-cure
Targis Dentin	TD	Ivoclar/Vivadent Liechtenstein	917433	Bis-GMA, UDMA, Decandiol DMA	Light-cure
Dentin/Enamel bonding resin	DB	Bisco IL, U.S.A.	099218	Bis-GMA, HEMA, Ketone	Light-cure
Special resin	SB	Jeneric/Pentron CT, U.S.A.	740941	Bis-GMA	Light-cure
Targis wetting agent	TW	Ivoclar/Vivadent Liechtenstein	926398	*r*-MPTS Ethanol	Self-cure
Targis Base 2	TB	Ivoclar/Vivadent Liechtenstein	A01197	Bis-GMA TEG-DMA	Light-cure

UHMWPE=ultra high molecular weight polyethylene;
DMA=dimethacrylate;
UDMA=urethane dimethacrylate;
PCDMA=polycarbonate dimethacrylate;
HEMA=hydroxyethyl methacrylate;
γ-MPTS=[γ-(methacryloxy)propyl]trimethoxysilane

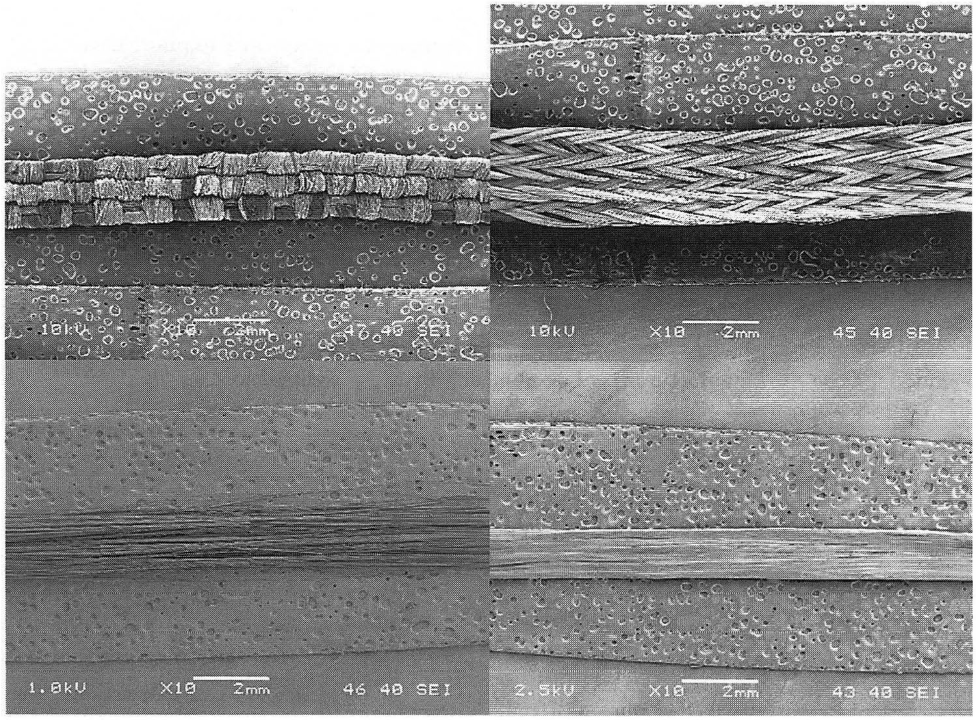

Fig. 1 Fibers used in the study (RB, GS, FK, and FF, clockwise from upper left).

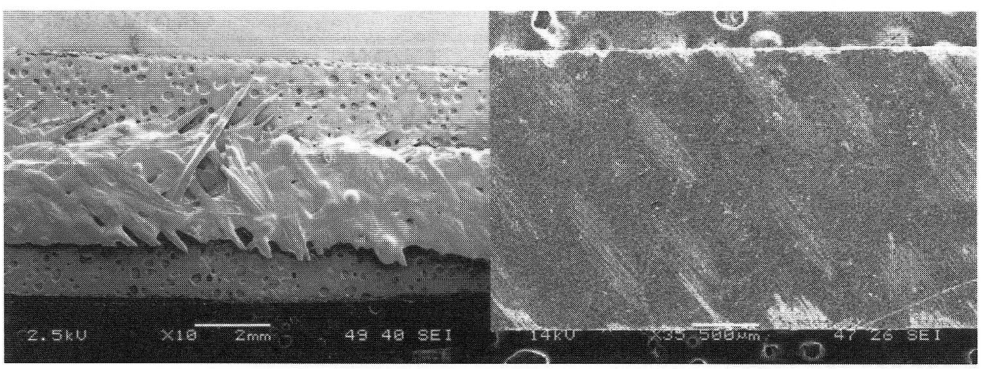

Fig. 2 SEM images of VF before curing (left) *vs.* VF after curing (right). Note that VF is the discontinuous and bi-directional type.

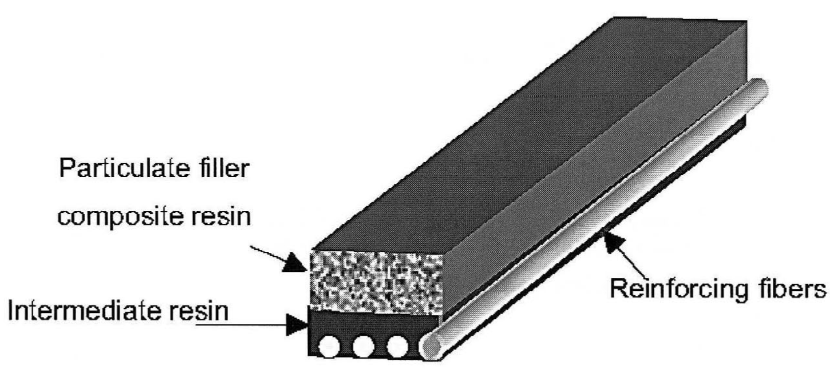

Fig. 3 Schematic diagram of a fiber-reinforced composite test specimen in the study.

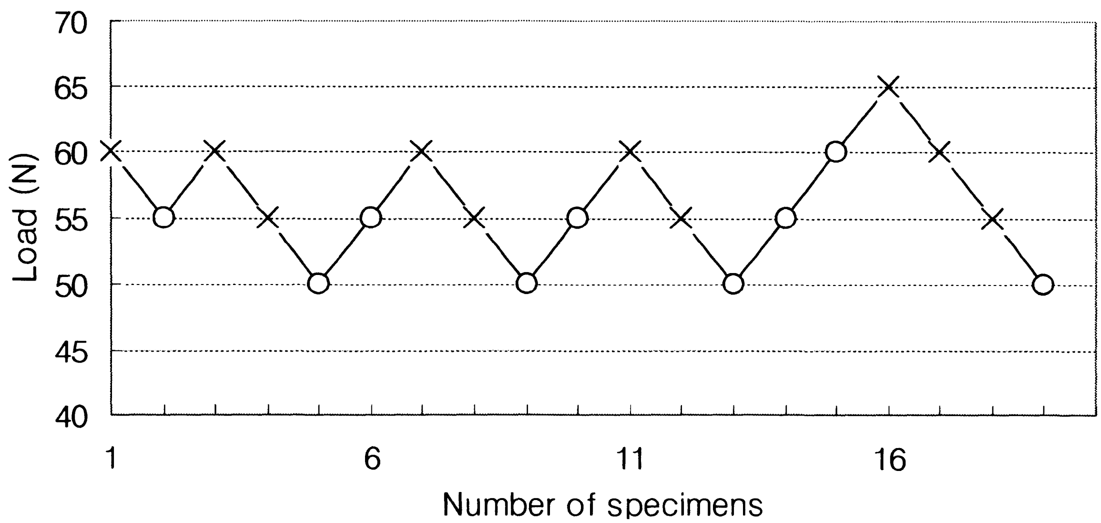

Fig. 4 Typical result of the staircase method to determine median fatigue strength at 10^5 cycles for GS+AF.
×: Fracture, ○: Non-fracture.

Table 2 Tabular Information Required for Analyzing Mean and Standard Deviation from Staircase Method (e.g., GS+AF)

Load (N)	i	Non-fracture	Fracture	ini	i^2ni
65			1	0	0
60	2	1	5	2	4
55	1	4	4	4	4
50	0	4		0	0
Sum		$N=\Sigma ni$ $=9$		$A=\Sigma ini$ $=6$	$B=\Sigma i^2ni$ $=8$

In case of RB+CB, RB+AF, GS+CB, and GS+AF, a total of 19 specimens were used, while 20 specimens were tested for the other groups.

was located in the mold (25 mm×2 mm×2 mm) to be the tension side of the specimen. For both FK and VF, which are preimpregnated in Bis-GMA, their own intermediate resin (IMR) and particulate filler composite resins were used, respectively: SB and SC (shade: A1) for FK; and TW followed by TB and TD for VF (shade: 210). In the case of the other fibers, DB was applied and cured for 20 seconds for interfacial treatment, and AF (shade: A1) was polymerized on it for 40 seconds with Curing Light XL 3000 (3M). In RB and GS, CB (shade: opaque), which could be used as a luting cement for fiber post, was additionally used. The non-reinforced specimens of particulate filler composite resins were made the same size as above, without fibers. All the specimens were made in the same manner as previously reported[7]. Twenty specimens of each group were prepared and stored in distilled water at 37℃ for 24 hrs before testing according to ISO 10477[9].

Fatigue test

A servohydraulic universal testing machine (EHF-F05, Shimadzu, Japan) was used to evaluate the fatigue strength in the three-point bending mode at a frequency of 5 Hz, until failure occurred or until 10^5 cycles had been completed in a water bath of 37 ℃. The fiber part of the specimen was located on the tension side with two supports at a distance of 20 mm.

The staircase method, with a stress increment of 2 N for composite resin and 5 N for FRC, except VF+TD (2.5 N load increment), was used to determine the fatigue strength. When the first specimen was broken at less than 10^5 cycles, another specimen was applied at a stress level one increment lower. If it was not fractured under a 10^5 cyclic load, another specimen was applied with a load one increment higher[14,15]. This procedure was continued, until twenty specimens of each group were expended. A typical result of the staircase method to determine median (mean) fatigue strength is shown in Fig. 4.

The calculation of the mean and standard deviation of the fatigue strength is determined via the staircase method by the following equations[14,15]

(Table 2).

$m=y'+d[A/N(+\text{or}-)1/2]$

$s=1.620\ d[(NB-A^2)/N^2+0.029]$ if $(NB-A^2)/N^2 \geq 0.3$

where, m=statistical estimate of mean fatigue strength

y'=lowest stress level at which the less frequent event occurred

d=step size, *i.e.* the stress increment

N=total number of less frequent events

s=statistical estimation of standard deviation

The plus sign (+) is used if the less frequent event is non-fracture and the minus sign (−) is used if the less frequent event is fracture.

Strain was calculated using the following equation[16]:

$$\varepsilon = \frac{6\delta h}{L^2}$$

where, ε is strain, δ is deflection, h is the thickness of specimen and L is the length of span. Obtained data of strain were analyzed by one-way analysis of variance (ANOVA) and by the Sheffé method for multiple comparison at the P=0.05 level.

Optical microscopic and SEM observations

After testing, the fractured aspects were evaluated by an optical microscope (Hi-Scope, KH-1000, Hirox Co., Ltd., Japan) at 120× magnification using a computer program (Image-Pro® Plus, Version 4.1, Media Cybernetics, U.S.A.). The fractured surfaces and the cross-sections embedded in epoxy resin were evaluated by a Field Emission Scanning Electron Microscope (JSM-6340F, JEOL DATUM, Japan) at 50 × and 3,000× magnifications.

RESULTS

Fatigue test

The dynamic fatigue strengths at 10^5 cycles and strains of particulate filler composite resins without (FF_0) and with reinforcing fibers (FF_r) are listed in Table 3. The fatigue strengths of the composite resins at 10^5 cycles were 49-57 MPa, while those of FRC were about 90-200 MPa. The fatigue strengths of glass fiber-reinforced composites were higher than other FRC, except for the VF-reinforced one which is a discontinuous and bi-directionally arranged fiber.

Table 3 Fatigue Strengths and Strains of Particulate Filler Composites with and without Reinforcing Fibers (Mean±Standard Deviation)

Resin	Fiber	Strain (%)		Fatigue strength (MPa)		(FF_r/FF_0) ×100(%)
		Resin	FRC	Resin, FF_0	FRC, FF_r	
CB	RB	0.4 ± 0.1^b	6.4 ± 2.0^a	48.8±7.0	167.7±15.1	344
	GS		1.1 ± 0.2^c		201.0±19.6	412
AF	RB	0.2 ± 0.1^c	5.9 ± 2.6^b	54.6±5.2	134.4±14.4	246
	GS		0.8 ± 0.0^c		209.4±14.4	384
	FF		0.9 ± 0.4^c		178.1±16.1	326
SC	FK	0.2 ± 0.1^c	0.5 ± 0.1^c	57.2±16.9	196.9±19.1	344
TD	VF	0.5 ± 0.2^a	0.8 ± 0.2^c	56.3±18.6	90.2±9.7	160

Means with the same superscripts are not significantly different at P<0.05.

Table 4 Fractured Aspects of Specimens after Repeated Loading

	No. of fractured specimens	Fiber fracture	Completely cut into two	*One part: adhered, The other: delaminated	**The middle: resin chopped out	Bending toward the direction of load
RB+CB	9	No			9	9
RB+AF	9	No			9	9
GS+CB	10	Yes	8	2		
GS+AF	10	Yes	10			
FF+AF	10	No		10		10
FK+SC	11	No		11		
VF+TD	12	Yes	12			

*One part of fiber adhered to the particulate filler composite resin, while the other part of fiber delaminated from it.

**The middle portion of particulate filler composite resin fractured out with a lot of cracks without the involvement of reinforcing fibers.

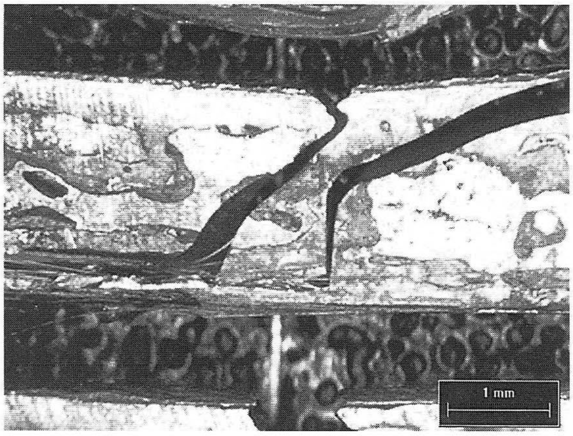

Fig. 5 Fractured aspect of FK+SC (original magnification ×120).

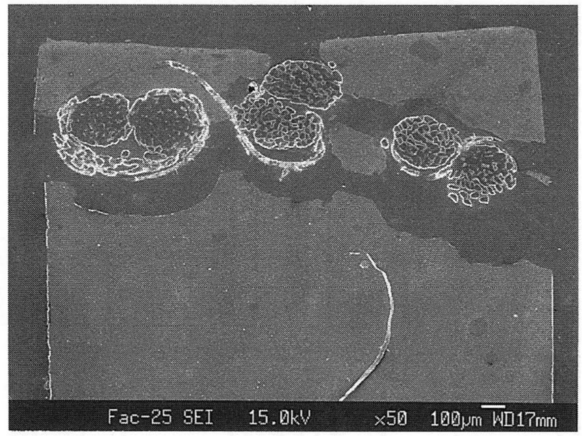

Fig. 6 Cross-section of RB+AF embedded in epoxy resin.

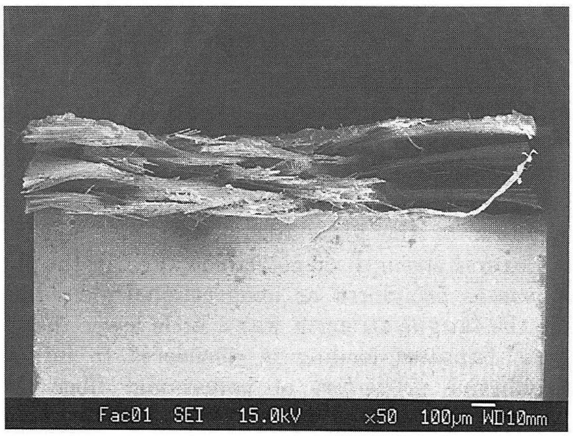

Fig. 7 Fractured surface of VF+TD in cross-section.

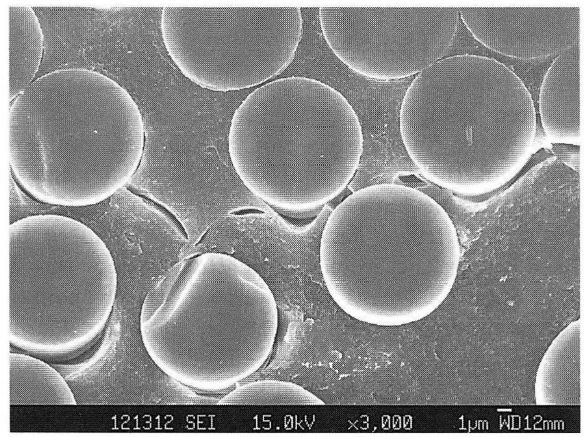

Fig. 8 Cross-section of FK+SC embedded in epoxy resin.

In terms of the reinforcing effects (FF_r/FF_0), the FRC with CB matrix showed a higher reinforcing effect than that with AF matrix in both RB- and GS-reinforced composites.

The strains of RB-reinforced composites showed the largest values and those of other FRC were not significantly different ($P<0.05$).

Optical microscopic and SEM observations

The fractured aspects of FRC after repeated loading were shown as an optical microscopic image (120×) in Fig. 5. Table 4 explains the fracture characteristics of fractured specimens based on the relationship between fiber and particulate filler composite resin. All the specimens of GS- and VF-reinforced composites were completely cut into two, including fibers. However, in RB-, FF-, and FK-reinforced ones, the fiber portions were sustained, although the overlying composite resins (the matrix) were broken in the middle.

In a cross-section of RB-reinforced composite embedded in epoxy resin, the fiber part was observed to be delaminated from the matrix after repeated loading (Fig. 6). In glass fiber-reinforced composites, fewer cracks were noted along the fibers than in the FF-reinforced one (Fig. 8).

Fig. 7 shows a representative SEM image of the fractured surface of FRC. GS- and VF-reinforced composites showed good adhesive properties between fiber and the matrix. For FF- and FK-reinforced ones, the adhered part of fiber to the matrix showed rather good adhesive properties, while most of the fibers were pulled out of the matrix in the delaminated part.

DISCUSSION

This study was carried out to evaluate the fatigue strengths at 10^5 cycles and the strains of particulate filler composite resins with and without reinforcing fibers, which could be a guide to the study using FRC. In most previous studies related to FRC, the specimens were made of fibers and the matrix with unfilled resin[17−19]. However, when FRC is applied to

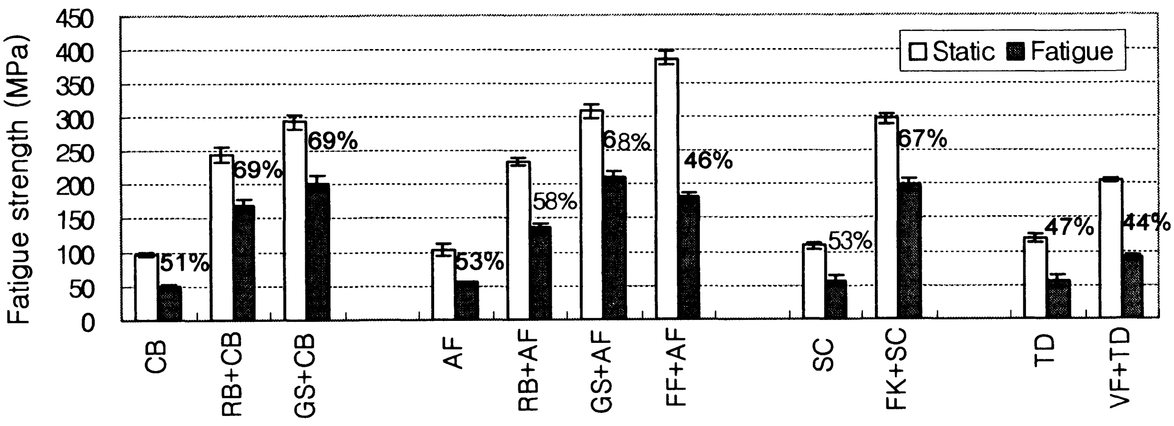

Fig. 9 Fatigue strength at 10^5 cycles *vs.* static ultimate flexural strength of particulate filler composite resin with and without reinforcing fibers.

the crown and bridge, the particulate filler composite resin is generally used. We adopted ceramic particle-reinforced composite resin for the matrix in this study. The fibers were not distributed uniformly throughout the matrix, but were located on the tension side of the matrix.

Many different fatigue testing procedures have been devised; the standard method using the *S-N* curve, constant stress level testing, the response or survival method (Probit method), step-test method, Prot method, staircase or up-and-down method, and the extreme value method[14]. Among these, the "staircase method" automatically concentrates testing near the mean and requires fewer tests[20,21], which is equally valid for determining the fatigue limit[14]. In particular, when $(NB\text{-}A^2)/N^2$ is larger than 0.3, the estimation of standard deviation becomes more accurate[14,15]. All the coefficients (0.47 for RB+CB, 0.62 for GS+CB, 0.44 for RB+AF, 0.44 for GS+AF, 0.53 for FF+AF, 0.60 for FK+SC, and 0.61 for VF+TD) were larger than 0.3 in this study.

According to Draughn[22], the fatigue strength of composite resin was applicable to 64% of compressive strength in the static test. The fatigue strengths of composite resins in this study occurred at stress levels relative to the yield flexural strengths of static test from previous study[7], as with polymers mentioned by Callister[1]. However, the fatigue strengths of FRC were about 60-70% of ultimate flexural strengths, except FF- and VF-reinforced composites, which were about 45% of the static values[7] (Fig. 9). The fatigue strengths of glass fiber-reinforced composites were higher than other fiber-reinforced composites except VF. Some critical fiber length is necessary for effective strengthening and stiffening of the composite material[1,23]. For this purpose, continuous fiber rather than discontinuous or short fiber is effective[24]. The strength of FRC is highly anisotropic, meaning that it had marked preference for any particular direction[2,23,25]. In this viewpoint of short fiber composition and bi-directional arrangement, VF is considered to result in low fatigue strength, in spite of the fair impregnated state confirmed by SEM (Fig. 2).

SEM observations indicated good aspects of impregnation of the glass fibers (GS, FK, and VF). The flexural strength of polyamide (FF) in the static test was as prominent as unidirectional glass fibers, while the fatigue strength was a little lower than the latter. Repeated loading is considered to influence the adhesive properties of polyaramid fiber much more than glass fibers. The lack of adhesion between UHMWPE (RB) fiber and IMR even after plasma spray treatment of the surface[5,17] seemed to be pronounced after repeated loading (Fig. 6), and it could be the cause of the lower fatigue value. For polymer-matrix composite, failure originates in or along the reinforcing fibers[23]. It is essential to get a high interfacial strength to maximize the overall strength of FRC[6,26,27]. Although it was suggested that polymer preimpregnation of fiber should have better impregnation[18,28,29), the differences between preimpregnation and non-preimpregnation in fatigue strengths were not recognized in this study, as previously reported[7].

The strain of RB-reinforced composites showed the largest values ($P<0.05$). If the strain of a material is large, despite its fatigue strength being rather high, it is difficult to apply it to dental use. The actual meaningful strengths are assumed to be lower than the evaluated fatigue strength, because a large deformation and cracks occurred before catastrophic failure.

In a previous study, we reported a method of determining fiber volume content[7]. For RB, GS, and FF, the weights of fibers were measured. For preimpregnated fibers, FK and VF, the weights of fibers were determined by combustion for 45 minutes at 700℃ and the fiber content as percentage by volume (V_f) (vol%) was calculated with the following formula[30]:

$$V_f=\frac{W_f/\rho_f}{W_f/\rho_f+W_r/\rho_r}\times 100$$

where W_f is the weight proportion of fiber, ρ_f is the density of fiber, W_r is the weight proportion of resin, and ρ_r is the density of resin. The fiber volume contents of the FRC were 9.07±0.34% for RB in CB, 8.60±0.26% for RB in AF, 6.33±0.50% for VF in TD, 4.14±0.06% for FK in SC, 3.96±0.08% for GS in CB, 3.92±0.13% for GS in AF, and 2.93±0.06% for FF in AF[7]. Because only a strip of each fiber as supplied by manufacturer was used in the study, the volume contents of RB and VF were larger than others. However, the volume contents between unidirectional glass fibers and polyaramid fiber were not much different. Further study is necessary to control the fiber contents to get more accurate results.

In the combination of reinforcing fiber and overlying particulate filler composite in this study, the interfacial adhesion and matching of flexural modulus of these two phases must have played an important role. To increase the mechanical properties of this kind of material, further research should be conducted to improve the interfacial bond strength. A new fiber-reinforced composite material, the hybrid, which means more than two kinds of fiber used in a single matrix in various arrangements, has been introduced to improve mechanical properties[1,23]. This hybrid type is considered to be necessary in the dental field, too. In combination of glass fibers with carbon or polyaramid fiber would be stronger and tougher, and it could increase the clinical success rate of FRC.

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