Reinforcement of Denture Base Resin with Short-rod Glass Fiber

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The present study investigated the feasibility of a high short-rod fiber content in denture base resins using polymethylmethacrylate (PMMA) powder with a small particle diameter. The effects of fiber contents on mechanical properties of composite resins were studied.

A commercial denture base PMMA powder (AC; average particle size, 150 μm) and an industrial PMMA powder (MB; average particle size, 4 μm) were selected. Short-rod glass fibers were mixed with the two powders at a mass ratio of $0-50$ %. Flexural strengths and moduli of the mixtures were evaluated by a three-point bending test.

The flexural strength of AC composites did not change regardless of fiber content, while that of MB composites increased significantly at fiber contents exceeding 40%. The flexural moduli of AC and MB composites at fiber contents exceeding 20% were significantly greater than those of AC and MB resins without short-rod glass fibers, respectively.

Keywords: Short-rod glass fiber, Flexural properties, Denture base resin

INTRODUCTION

Polymethylmethacrylate (PMMA) has been widely used as a denture base material. Several reasons contribute to its widespread use: economical, stable in the oral environment, and easily constructed and repaired. However, mechanical failures of PMMA dentures often occur[1]. Therefore, many researchers have attempted to improve the mechanical properties of denture base resins[2-6].

The addition of different types of fiber, such as carbon[2], aramid[4], polyethylene[3,5], and glass[6] fiber products, has been evaluated to overcome the physical and mechanical limitations of denture base resins. Carbon and aramid fibers are not practical because of polishing difficulties and their undesirable esthetic appearance[6]. The mixture of polyethylene fibers and PMMA powder requires a complicated surface treatment and does not significantly improve the mechanical properties of denture base resins[5]. Therefore, much attention has been directed toward the glass fiber due to its excellent esthetic appearance and the absence of cytotoxicity[8].

Two types of glass fibers are used for reinforcing denture base resins; one is a continuous fiber similar to a woven sheet or having a stick-shape[9-14], while the other is short-rod glass fiber used as a filler[15-19]. The former has been widely investigated and is accepted as providing high strength and stiffness in a direction parallel to the fibers. However, it is difficult to construct, further technical procedures are required to orientate the continuous fibers to reinforce dentures at weak regions[1,3]. Moreover, Vallittu[10] indicated that the use of continuous fibers for denture reinforcement resulted in the formation of voids inside the PMMA resin. On the other hand, short-rod glass fibers in composites provide similar properties in all directions. As a result, their composites are relatively isotropic[12] and are easily used with conventional compression molding techniques. However, reinforcement with short-rod glass fibers has not been widely investigated as compared to continuous fibers.

Stipho[15] reported that PMMA composites with a short-rod glass fiber content of 1% provided the best flexural strength, and that more than 5% short-rod glass fiber content did not improve the mechanical properties of denture base resins. A higher glass fiber content showed that the fibers tended to clump together when mixed with a polymer, and that porosity was increased due to formation of void spaces in the composites[15]. Therefore, earlier studies have suggested on the use of a low content of short-rod glass fibers[17,19].

Generally, flexural strength and modulus generally increase with increasing volumetric content of glass fibers[7]. To improve mechanical properties of PMMA composites, uniform PMMA composites with a high short-rod glass fiber content without clumping of fibers and voids are therefore desirable.

Özdeír and Polat[19] reported that flexural strength of an injection-molded resin with 5 mass% short-rod fibers increased, and contained less void spaces compared to a compression-molded resin. Generally, the particle size of PMMA powder used in the injection molding technique is smaller than that in the compression molding technique. Therefore,

the choice of not only the molding method but also the PMMA powder particle size might help eliminate void spaces. The particle size of commercial denture PMMA powder is generally more than 25 μm. However, the diameters of common glass fibers range from 8 to 20 μm which is smaller than the particle size of PMMA powder. On this account, PMMA powder might prevent uniform distribution of short-rod glass fibers and create void spaces.

The present study aimed to investigate the feasibility of a high content of short-rod fibers in denture base resins using PMMA powder with a small particle diameter, and evaluate the effects of a high fiber content on mechanical properties.

MATERIALS AND METHODS

Materials

The materials used in this study are listed in Table 1 and their SEM images are shown in Fig. 1. Two types of PMMA powder were selected: one was a commercial denture base PMMA powder (Acron Clear, GC Corp., Tokyo, Japan; average particle size, 150 μm; AC), while the other was an industrial PMMA powder (MB-4C, Sekisui Kasei, Osaka, Japan; average particle size, 4 μm; MB). AC contained benzoyl peroxide, but MB did not.

Short-rod glass fibers with an aspect ratio of approximately 20 (Milled E-glass fiber, Asahi Fiber Glass, Tokyo, Japan; average length, 200 μm; average diameter, 10 μm) were selected. The glass fibers

were silanated with -methacryloxypropyltrimethoxy-silane (KBM503, Shin-Etsu Chemical, Tokyo, Japan)[20].

PMMA powder and silanated glass fibers were mixed at mass ratios of 0, 10, 20, 30, 40, and 50 mass using a sun-and-planet mixer (NBK-1, Nihon Seiki, Nagano, Japan). Methyl methacrylate (MMA-HQ-S, Mitsubishi Rayon, Tokyo, Japan) with and without 2 mass of benzoyl peroxide (SAJ class 1, Sigma-Aldrich Japan, Tokyo, Japan) was used for dough processing of MB and AC, respectively.

Flexural properties

A rectangular pattern of each specimen ($64.0\times 10.0\times 3.5$ mm) was prepared, and the dough mixture at a P/L ratio of 1.66 g/mL was packed and polymerized using the conventional compression mold technique at 60 for 60 minutes, and 100 for 30 minutes. Following removal from the mold, specimens were polished with 600 SiC paper under running water so that their final size was $64\times 9.9\times 3.3$ mm. Specimens were then stored in 37 distilled water for 50 hours. Six specimens for each group were prepared.

Specimen sizes were measured with a micrometer (293-421-20, Mitsutoyo, Kanagawa, Japan; minimum reading, 0.001 mm) before flexural testing. A three-point bending test was performed with a support span distance of 50 mm and at a crosshead speed of 5 mm/min using a universal testing machine (5500R, Instron, Canton, MA, USA) at room temperature. Flexural strengths and moduli were calculated from

Table 1   Materials used in the present study

Material	Product Name	Manufacturer	Lot no.
E-glass fiber	Milled Fiber	Asahi fiber glass	MF20JH1-20
PMMA powder	Acron Clear	GC	0109281/0309102
PMMA powder	MB-4C	Sekisui Kasei	DB-8326
Methyl methacrylate	MMA-HQ-S	Mitsubishi Rayon	241716
Benzoyl peroxide	SAJ class 1	Sigma Aldrich	A8345
-methacryloxypropyltrimethoxysilane	KBM503	Sin-Etsu Chemical	11193

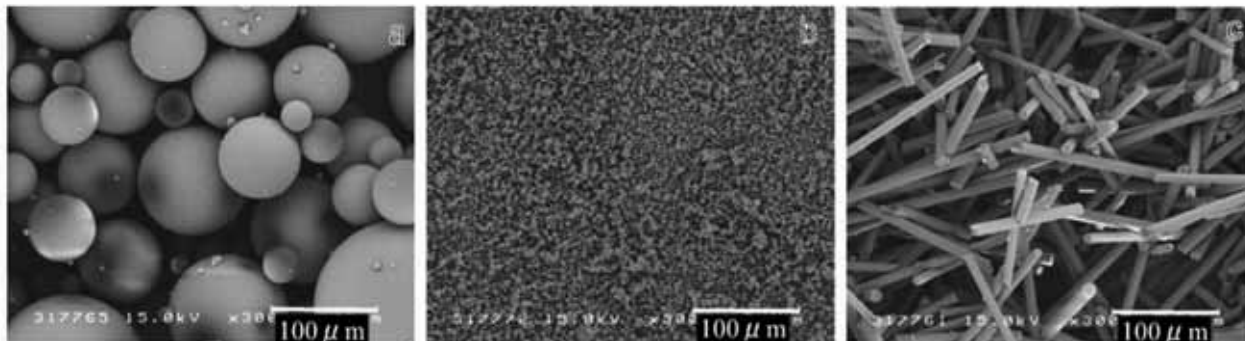Fig. 1   SEM images of the materials used: (a) A commercial denture base polymer of 150 μm φ; (b) An industrial PMMA powder of 4 μm φ; (c) Short-rod glass fibers of 10 μm $\varphi \times$ 200 μm.

the following equations using statistical software (Series IX, Instron):

$$\sigma = 3Fl/2bh^2$$

$$E = F_l l^3/4bh^3 d$$

where F is the maximum load (N), l is the width of the support span (mm), b is the width (mm) of the specimen, h is the height(mm) of the specimen, F_l is the load (N) at a point in the straight-line portion of the trace, and d is the deflection (mm) at load F_l.

Scanning electron microscopic (SEM) observations
Specimens from each group were carbon-coated to examine both polished and fracture surfaces using a scanning electron microscope (S-4500, Hitachi, Tokyo, Japan).

Statistical analysis
Mean values and standard deviations of the data for each group were calculated. Data were analyzed using two-way analysis of variance (ANOVA) and Tukey's HSD multiple comparison test with a significance level of 5 using statistical software (JMP IN ver 5.0, SAS, Cary, NC, USA).

RESULTS

Figure 2 shows the specimens after flexural test. Glass fibers of all specimens, except for F30, F40, and F50 of the AC composites, were seen as uniformly distributed by naked eye.

Figures 3 and 4 show the flexural strengths and moduli with standard deviations, respectively. Flexural strengths of AC and MB resins without glass fiber were 106.7 and 102.7 MPa, respectively. The flexural strength of AC composites decreased with increasing glass fiber content. At F50, the flexural strength of AC composite was 88.2 MPa.
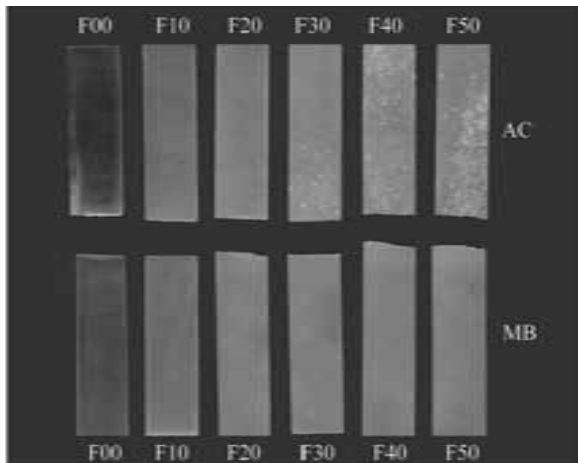

Fig. 2   Specimens after flexural testing.

However, the flexural strength of MB composites increased with increasing glass fiber content. At F50, the flexural strength of MB composite was 136.2 MPa. Two-way ANOVA indicated that PMMA type and fiber content did not significantly affect flexural strength. However, interaction between PMMA type and fiber content was a significant factor. Tukey's HSD analysis showed that the flexural strengths of MB composites of F40 and F50 were significantly greater than that of MB resin; however, the flexural strengths of AC composites were not significantly different regardless of fiber content. When comparing AC and MB composites with the same fiber content, the flexural strengths of MB composites were significantly higher than those of AC composites of F30, F40, and F50.

Flexural moduli of AC and MB resins without glass fiber were 2.8 GPa and 2.8 GPa, respectively.
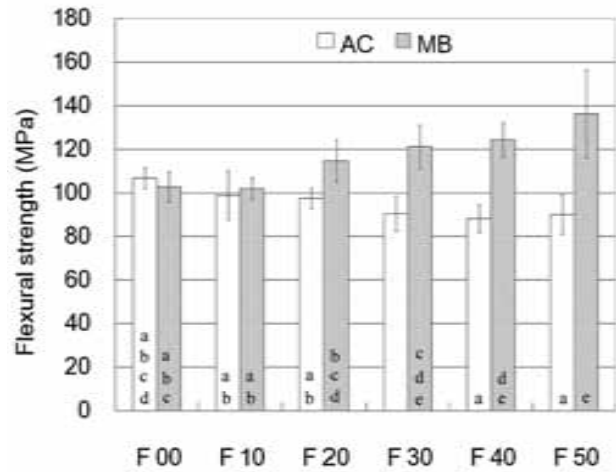

Fig. 3   Flexural strengths (means with standard deviations). Bars with the same letters are not significantly different (P<0.05).
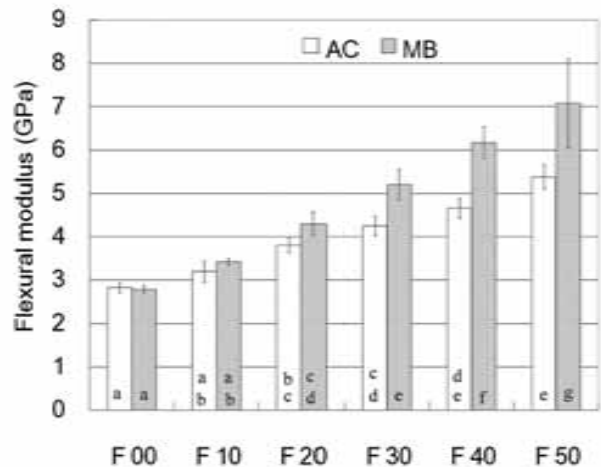

Fig. 4   Flexural moduli (means with standard deviations). Bars with the same letters are not significantly different (P<0.05).
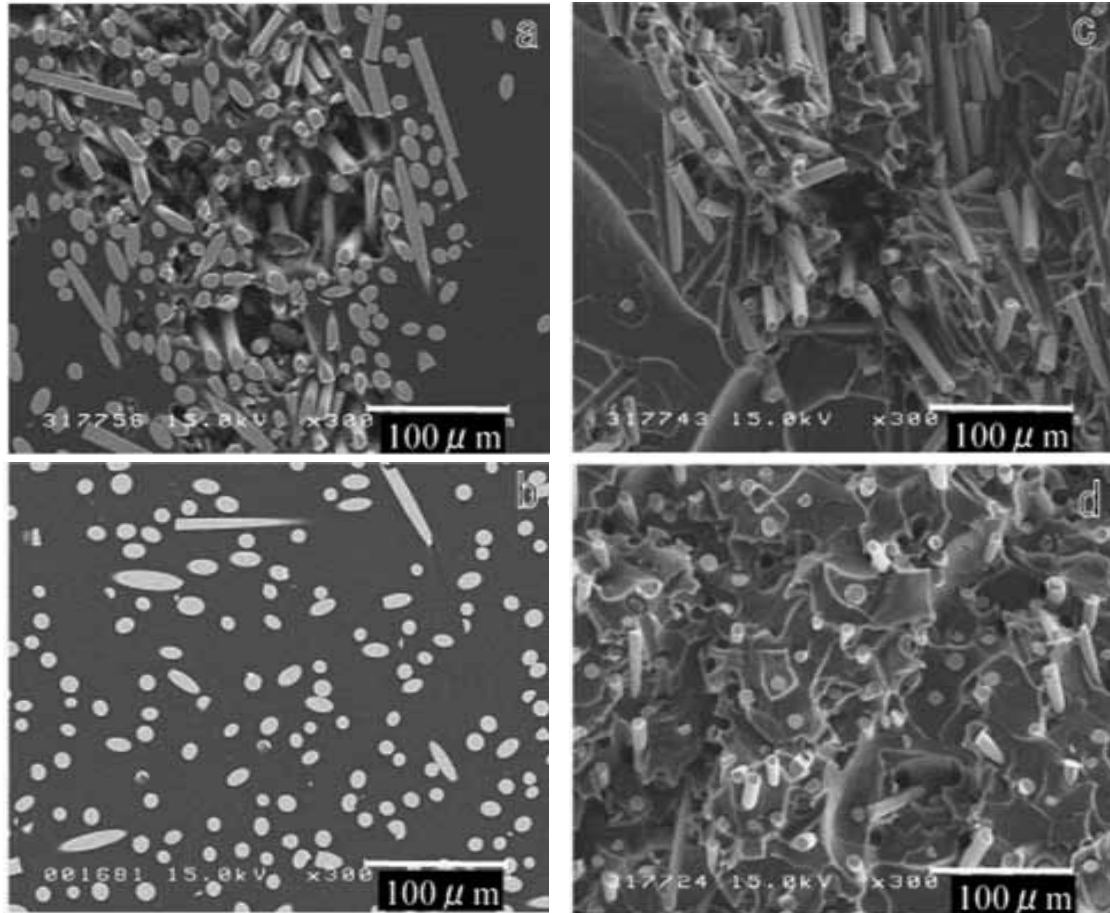
Fig. 5   SEM images of polished and fractured surfaces of AC (a, c) and MB (b, d) composites with a short-
        rod glass fiber content of 40 mass .

The flexural modulus of AC composite of F50 was 5.4 GPa, while that of MB composite was 7.1 GPa. The flexural moduli of both AC and MB composites tended to increase with increasing glass fiber content.   Two-way ANOVA indicated that PMMA type was not a significantly effective factor in flexural modulus.   However, fiber content and interaction between PMMA and fiber content were significantly effective factors.   The effect of fiber content was greater on MB than on AC.   Tukey's HSD analysis suggested that flexural moduli of AC and MB at fiber contents exceeding 20   were significantly greater than those of AC and MB resins, respectively.   When comparing AC and MB composites with the same fiber content, the flexural moduli of MB composites of F30, F40, and F50 were significantly higher than those of AC.

SEM images of the polished surface of F40 are shown in Figs. 5a and b.   Agglomeration of fibers and void spaces inside the agglomerated fibers were observed in AC composite (Fig. 5a).   However, short-rod glass fibers seemed to be uniformly distributed in MB composite (Fig. 5b).   With regard to the fractured surfaces, agglomeration of fibers and void spaces were seen in AC composite (Fig. 5c) but not in MB composite (Fig. 5d).

DISCUSSION

It is well known that the effects of short-rod fiber reinforcement are controlled by the amount, distribution, diameter, and aspect ratio of the glass fiber used[21].   Regarding glass fibers and resin composites, the length of a glass fiber is an important factor that determines the mechanical characteristics of a composite material[21].   This is because of the great discrepancy between the flexural moduli of glass fibers and matrix resin.   The minimum fiber length for improving strength of a composite material is called the critical length (lc), which can be calculated from the following formula[16]:

$$lc = \sigma_{tf}D/2t$$

where σ_{tf} is the tensile strength of the glass fiber (MPa), D is the diameter of the glass fiber (mm), and

t is the shear strength of the matrix resin (MPa).

When σ_{tf} is 2200 MPa[16], D is 10 μm, and t is 122 MPa[22], lc was estimated to be 90 μm. Therefore, the length of the short glass fiber used in the present study (200 μm) was greater than the critical length. Longer glass fibers will greatly improve the mechanical properties of composite materials[21]. However, longer glass fibers might also cause difficulties for polishing and mixing without forming void spaces. On this note, future research is necessary to determine an appropriate and optimal fiber length to improve the mechanical properties of denture base resins.

SEM images showed that short-rod glass fibers were uniformly distributed in MB but not in AC composites. The particle size of MB was 4 μm, which was smaller than the glass fiber diameter. The particle size of AC was 150 μm, which was greater than the glass fiber diameter and similar to the glass fiber length. Therefore, the average particle size of PMMA powder was an important factor to assure uniform mixing with the glass fibers.

Flexural strength of MB composite in the presence of 50 short-rod fibers increased by 33 compared to without short-rod fibers. However, the flexural strength of AC composites did not increase but rather decrease with increasing fiber content. Solnit[9] reported that flexural strength increased by 10.8 with 0.1 g of silanated short-rod glass fiber (polymer/monomer ratio was 27 gm/15 ml in their samples.). Stipho[15] also indicated that flexural strength increased by 18 by adding 1 of silanated 2-mm short-rod glass fiber. Similarly, Chen et al.[17] reported that flexural strength increased by 8 with 2 of unsilanated 4-mm short-rod glass fiber. The increase in flexural strength in the present study was greater than those of the above-mentioned studies. The PMMA powders used in previous studies were commercial products for denture bases. Therefore, particle size of the powder was greater than the glass fiber diameter. As a result, there was great difficulty for uniformly mixing and void spaces were thus created inside the composites.

A high flexural modulus of denture base materials is an important requirement to resist denture deformation during mastication[1]. However, few studies have discussed on flexural modulus improvement using short-rod fiber reinforcement. Flexural moduli of both AC and MB composites increased with increasing glass fiber content, and the flexural modulus of MB composites was substantially increased compared to that of AC composites. Flexural moduli of AC and MB composites in the presence of 50 short-rod glass fiber content increased by 154 and 93, respectively, as compared to those without short-rod glass fiber. Differences between the two composites could be the result of clustered fibers and void formation in AC.

Vallittu[10] indicated that poor wetting of the fibers might explain void spaces in acrylic glass fiber composites. In addition, Özdemír and Polat[19] indicated that there were more void spaces in composites made with a compression molding technique as compared to those with an injection molding technique; moreover, high glass fiber contents could easily result in void spaces. However, in the present study, no obvious void spaces were observed in MB composites even with 50 mass content of glass fiber. In other words, the size of MB powder might have been responsible for eliminating void formation and contributing to the uniform distribution of glass fibers.

The present study demonstrated that the thickness of the denture base could be reduced without reducing or compromising fracture strength when short-rod fibers were used. Thinner denture base materials are desirable for good gustatory response and patient satisfaction. Moreover, decreases in water sorption and solubility of denture base resins containing short glass fibers are advantageous for clinical use[14].

A considerable disadvantage of using short fiber was the presence of protruding ends in the finished specimens, which might show a rough surface. If the glass fiber surface was appropriately silanated, the denture surface could be polished to a smooth finish with conventional polishing procedures, as shown in Figs. 5a and b. Waltimo et al.[11] reported that an increase in the adherence of *Candia albicans* was not observed on the polished surface of a denture base polymer with E-glass reinforcement.

Using small-particle-size PMMA powder reinforced with a higher content of short-rod glass fiber significantly improved the mechanical properties. However, the relationship between PMMA powder particle size and glass fiber length was not fully investigated in the present study. Thus, a further research is necessary to clarify this issue.

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