Effect of fiberglass orientation on flexural properties of fiberglass-reinforced composite resin block for CAD/CAM

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A fiberglass-reinforced composite resin (FRP) block using a plain woven fiberglass sheet for CAD/CAM has been introduced in dental practice. The aim of the present study was to evaluate the effects of the fiberglass sheet orientation on the flexural properties of an FRP block. The flexural properties of five types of fiberglass sheet-assigned specimens were examined using a three-point bending test. A one-way analysis of variance revealed that the orientation of fiberglass sheet significantly influenced the flexural strength, 0.2% yield strength, and flexural modulus. The values of the flexural properties of the FRP were the largest when the fiberglass sheets were perpendicular to the applied force, and the smallest when the fiberglass was parallel to the same. The flexural properties of the FRP block were anisotropic and they were significantly influenced by the orientation of fiberglass sheet.

Keywords: FRP block for CAD/CAM, Fiberglass orientation, Flexural strength, Flexural modulus

INTRODUCTION

In recent years, the development of alternative materials for dental prostheses without metal has been actively pursued owing to increasing prices of the noble alloys and patients' fear of allergic reactions to metals.

The conventional alternative to metal is a composite resin. Fiberglass is also suitable for such applications because it is highly translucent and has a high tensile strength and a low density. Therefore, a sheet or bundle of unidirectional fiberglass covered with composite resin was applied for dental restorations. Even though the advantage of composite resin with fiberglass, there are problems such as interlayer delamination caused by water absorption and possible mechanical irritation caused by exposed fiberglass.

Restorations using composite resin with fiberglass are relatively lighter compared to conventional metal restorations. They are also preferable with regard to esthetics owing to their translucency and seemingly natural appearance. However, such restoration requires skillful technique and its fabrication is a complicated process. Fractures of the pontic of fixed partial dentures were reported¹⁻⁴, and the reinforcement of the pontic was suggested^{5,6}. A composite resin prosthesis with fiberglass is considered unsuitable for long-span prosthesis, that is, those with two or more pontics⁷.

Due to advancement of the CAD/CAM technology, a fiberglass-reinforced composite resin (FRP) block for CAD/CAM has been introduced in dental practice. This FRP block consists of layered sheets of plain woven fiberglass. There are several woven patterns of

fiberglass sheets. The plain weave is firm and strong and has a high wear resistance. It has some spaces at the strand crossing points; therefore, it is relatively thick with a high permeability. The twill weave is elastic and hard to wrinkle, but it has a poor durability and wear resistance. On the surface of the satin weave, only the warp or woof is exposed; it is soft and glossy but has a poor wear resistance. The FRP block has been claimed to be applicable for use in a single crown, fixed partial denture, and superstructure of implant-supported dentures⁸⁻¹¹. The mechanical properties of the FRP block were reported to be anisotropic and they were influenced by several factors such as the composition and diameter of the fiberglass^{12,13}; amount, direction, and woven pattern of the fiberglass sheet^{14,15}; properties of the base resin^{16,17}; and effectiveness of the silane coupling agent¹⁸⁻²⁰. However, there is no previous report on the mechanical properties of such a commercial FRP block. The aim of the present study was to evaluate the effects of the orientation of a fiberglass sheet on the flexural properties of an FRP block. The null hypothesis was that the fiberglass sheet orientation did not influence the flexural properties of the FRP block.

MATERIALS AND METHODS

Materials used

The materials used in the present study are summarized in Table 1. The FRP block was disk-shaped and it consisted of plain woven sheets of E-glass fiber and epoxy resin. A hybrid-type composite resin for veneering was used as a reference.

Table 1 Materials used in this study

Type	Trade name	Components	Manufacturer	Lot. No
FRP Block	Trinia ø98×25 mm	Glass fiber, Epoxy resin	Bicon	04081114
Composite Resin	Ceramage A2B	UDMA, Zirconium silicate, Colorant, others	Shofu	051598

Fig. 1 Schematic of top and lateral views of test specimens, orientation of woven fiberglass sheet, and direction of load (arrow). The letters are abbreviations that indicate the type of fiberglass sheet orientation.

Flexural properties

A 2.0-mm-thick FRP block was prepared parallel to a woven sheet using an automatic precision cutting machine (Secotom-50, Struers, Cleveland, OH, USA). Then, a 2.0×20×25-mm plate-shaped block was prepared in which the fiberglass was set either parallel (+) or at 45° (x) to the long axis using the cutting machine. Another 2.0-mm-thick rectangular block was obtained by slicing the FRP block vertical to the woven sheet. Then, a 2.0×20×25-mm plate was prepared to set the woven sheet vertical (|) to its long axis by using the cutting machine. A 2.0×2.0×25 mm bar-shaped specimen was fabricated from the plate-shaped block by using a low-speed cutting machine (Isomet, Buehler, Lake Bluff, IL, USA) and polished using a polishing machine (ML-150P, Maruto, Tokyo, Japan) with an SiC grinding sheet of P600. Finally, 30 bar-shaped specimens with three different fiberglass directions were prepared; 12 specimens had the '+' pattern, 12 specimens had the 'x' pattern, and 6 specimens had the '|' pattern.

A hybrid-type composite resin was squeezed into a 2.2×30×25-mm mold on a glass plate and covered with another glass plate. A rectangular block was obtained from the mold after photo polymerization for 180 s. A

2.0×2.0×25-mm bar-shaped specimen was fabricated in the same manner as mentioned above (CR).

After storage in a dry desiccator for at least one week, a three-point bending test was performed at a crosshead speed of 0.5 mm/min and a support span width of 20.0 mm by using a universal test machine (AG-X plus 50 kN, Shimadzu, Kyoto, Japan). The flexural strength, 0.2% yield strength, and flexural modulus were calculated using materials testing software (Trapezium X, Shimadzu). For the '+' and 'x' specimens, the load was applied parallel or perpendicular to the woven sheet; specimens to which a load was applied on the '+' surface and perpendicular to the '+' surface were classified as '+' type and '-+' type, respectively; specimens to which a load was applied on the 'x' surface and vertical to the 'x' surface were classified as 'x-' type and '-x' type, respectively. The '|' specimens, to which a load was applied parallel to the woven sheet, were classified as "||" type specimens for evaluation of flexural properties. Therefore, five types of bar-shaped specimens were evaluated with differing fiberglass sheet directions (Fig. 1).

Inorganic filler content

The inorganic filler contents of the FRP block and CR were measured with an ash method following ISO 1172:1996²¹⁾. After the bending test, specimens of the ‘+–’ type were used for filler content measurement. An alumina crucible (PC2, Nikkato, Osaka, Japan) was heated in an electric furnace (Burn Out Furnace 007EX, KDF, Kyoto, Japan) at 625°C for 30 min. The weight of the crucible was measured using a precision digital balance (AUW120D, Shimadzu; minimum reading: 0.01 mg) after it was cooled to ambient temperature in a desiccator. This measurement was repeated until a constant mass was obtained in two subsequent measurements. Approximately 1.0 g of the ‘+–’ type specimen was placed in the crucible, and the weight of each specimen including the crucible was measured with the precision digital balance. The crucible containing the specimen was heated in an electric furnace, as detailed above, to burn out the organic components. After cooling to ambient temperature in a desiccator, the residue and crucible were reweighed until a constant mass was obtained. The inorganic filler content (mass%) was determined using the following equation:

$$\text{Filler content} = (m_3 - m_1) / (m_2 - m_1) \times 100$$

where m_1 is the mass of the dry crucible, m_2 is the mass of the dry crucible and the dried specimen, and m_3 is the mass of the crucible and the residue after calcination. The inorganic filler content of the CR was also determined. Five measurements for each material were performed.

Statistical analysis

The flexural strength, 0.2% yield strength, and flexural modulus were analyzed using one-way analysis of variance and a Tukey multiple comparison test using statistical software (JMP Ver.11, SAS institute, Cary, NC, USA) at a significance level of 5%.

RESULTS

The bar-shaped specimen did not completely fracture except for the ‘||’ type and CR. Typical CCD microscopic images of the specimens after the bending test are shown in Fig. 2. Cracks were observed on the tensile side of the ‘+–’ and ‘–+’ specimens, but pullout of the fiberglass was observed on the tensile side of the ‘–×’ specimens. The

Fig. 2 CCD microscopic images of specimens after three-point bending test.

Table 2 Effect of fiberglass sheet orientation on flexural strength, 0.2% yield strength, and flexural modulus

	Fiberglass	Flexural strength	0.2% Yield strength	Flexural modulus
	orientation	(MPa)	(MPa)	(GPa)
FRP	+–	390±19 ^A	379±30 ^E	15.9±0.3 ^I
	–+	406±19 ^A	397±14 ^E	17.0±0.3 ^H
	×–	187±4 ^B	82±2 ^G	8.9±0.5 ^K
	–×	179±3 ^B	82±2 ^G	9.6±0.3 ^J
		101±4 ^D	65±23 ^G	6.9±0.1 ^L
CR		156±9 ^C	121±1 ^F	10.3±0.3 ^J

mean±SD, $n=6$

Values with the same superscript letter were not significantly different ($p>0.05$)

cracks propagated inside the resin layer between the fiberglass sheets.

The values of flexural strength, 0.2% yield strength, and flexural modulus are summarized in Table 2. The flexural strengths of the FRP block ranged from 101 MPa (||) to 406 MPa (—+), and that of the CR was 156 MPa. The flexural strengths differed significantly; they were ranked as follows: || < CR < —x = x < + = —+. The 0.2% yield strengths of the FRP block ranged from 65 MPa (||) to 397 MPa (—+), and that of the CR was 121 MPa. The 0.2% yield strengths also differed considerably; they were ranked as follows: || = —x = x < CR < + = —+. The flexural moduli of the FRP block ranged from 6.9 GPa (||) to 17.0 GPa (—+), and that of the CR was 10.3 GPa. The flexural moduli also differed significantly; they were ranked as follows: || < x < —x = CR < + < —+.

The inorganic filler mass contents and the standard deviations of the FRP block and CR were 53.9±1.2% and 66.3±0.1%, respectively.

DISCUSSION

The null hypothesis that the fiberglass sheet orientation did not influence the flexural properties of the FRP block was rejected.

The FRP block used consisted of plain woven sheets; therefore, the epoxy resin could penetrate through the strand crossing points of the woven fiberglass sheets. Exposure of the glass fiber is not desirable owing to the anisotropic nature and solubility of the fiberglass; therefore, the FRP block is often veneered with a composite resin to modify its appearance and cover the exposed fiberglass. The composite resin for veneering recommended by the manufacturer was used as the reference in the present study.

A three-point bending test is commonly used for evaluating the mechanical properties of polymer-based materials. The procedures of the bending test complied with ISO 4049:2009 —Dentistry— Polymer-based restorative materials. The test specimen was assigned based on the direction of the woven sheets.

The specimens were stored in a dry desiccator for at least one week to remove any moisture. The effects of storage in water on the mechanical properties will be studied in the future.

The flexural strength, 0.2% yield strength, and flexural modulus varied with the fiber direction. When a load was applied perpendicular to the fiber direction such as for the +— and —+ type specimens, the flexural strength, yield strength, and flexural modulus were high. However, when a load was applied parallel to the fiber direction such as for the || type, the values of these properties were the lowest.

These results are reasonable because the load was applied directly to the fiberglass in the case of the +— and —+ type specimens. The flexural strength of the || type specimen was less than that of the referenced CR. This value was considered as the flexural strength of epoxy resin. The flexural modulus increased proportionally with the volumetric content of the fiberglass, which is

referred to as the mixture law. The mass filler content of the FRP was 53.9%. The volumetric fiberglass content was calculated as 35.6% when the density of the epoxy resin and E-glass were assumed to be 1.2 and 2.55 g/cm³, respectively. This fiberglass content was greater than the reported values of FRP products, which are generally 2.9 to 9.1%²²⁾. The flexural modulus of FRP containing 35.6 vol% fiberglass was calculated as 29.0 GPa, when the flexural modulus of the epoxy resin and E-glass were assumed to be 2.0 and 77 GPa, respectively. However, plain woven sheets were used for the FRP block in the present study; therefore, almost half of the fiberglass was parallel to the specimen's long axis. As a result, the flexural modulus was calculated as 14.5 GPa, which is compatible with the measured values of the +— and —+ specimens.

The flexural strengths and flexural moduli of the FRP products were reported to be 203 to 386 MPa and 5 to 15 GPa, respectively²³⁾; however, fixed partial dentures using these FRP products need to be subjected to further experimental study. The FRP block used in the present *in vitro* study was strong and stiff compared with other FRP products. The flexural strength of the FRP block was approximately 400 MPa when the load was applied perpendicular to the direction of the fiber. The flexural strength was approximately 100 MPa when the load was applied parallel to the direction of the fiber. The manufacturer claims that such an FRP block can be used for a single crown, fixed partial denture, and superstructures of implant-supported dentures.

The flexural properties in the present study were determined *in vitro* and the effect of water was not considered. Therefore, further studies are necessary to evaluate the durability of the mechanical properties of the FRP block, determine the bonding between the veneering composite resin and the FRP block, and perform a clinical evaluation of FRP block restorations.

CONCLUSION

Considering the limitations of this *in vitro* study, the following conclusions were drawn. The flexural properties of the FRP block were significantly influenced by the orientation of fiberglass sheets. The flexural strengths of the FRP block varied from 101 to 406 MPa. The flexural modulus of the FRP block varied from 6.9 to 17.0 GPa.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

REFERENCES

- 1) Kolbeck C, Rosentritt M, Behr M, Lang R, Handel G. In vitro examination of the fracture strength of 3 different fiber-reinforced composite and 1 all-ceramic posterior inlay fixed partial denture systems. *J Prosthodont* 2002; 11: 248-253.
- 2) Pfeiffer P, Grube L. In vitro resistance of reinforced interim fixed partial dentures. *J Prosthet Dent* 2003; 89: 170-174.
- 3) Ozcan M, Breuklander MH, Vallittu PK. The effect of box preparation on the strength of glass fiber-reinforced composite

- inlay-retained fixed partial dentures. *J Prosthet Dent* 2005; 93: 337-345.
- 4) Dyer SR, Sorensen JA, Lassila LV, Vallittu PK. Damage mechanics and load failure of fiber-reinforced composite fixed partial dentures. *Dent Mater* 2005; 21: 1104-1110.
 - 5) Xie Q, Lassila LV, Vallittu PK. Comparison of load-bearing capacity of direct resin-bonded fiber-reinforced composite FPDs with four framework designs. *J Dent* 2007; 35: 578-582.
 - 6) Perea L, Matinlinna JP, Tolvanen M, Vallittu PK. Fracture behavior of pontics of fiber-reinforced composite fixed dental prostheses. *Dent Mater J* 2015; 34: 746-753.
 - 7) Freilich MA, Meiers JC, Duncan P, Goldberg AJ. Fiber-reinforced composites in clinical dentistry. Illinois: Quintessence Publishing Co; 2000. p. 26-27.
 - 8) Başaran EG, Ayna E, Vallittu PK, Lassila LV. Load bearing capacity of fiber-reinforced and unreinforced composite resin CAD/CAM-fabricated fixed dental prostheses. *J Prosthet* 2013; 109: 88-94.
 - 9) Bonfante EA, Suzuki M, Carvalho RM, Hirata R, Lubelski W, Bonfante G, *et al.* Digitally produced fiber-reinforced composite substructures for three-unit implant-supported fixed dental prostheses. *Int J Oral Maxillofac Implants* 2015; 30: 321-329.
 - 10) Seemann R, Marincola M, Seay D, Perisanidis C, Barger N, Ewers R. Preliminary results of fixed, fiber-reinforced resin bridges on four 4- × 5-mm ultrashort implants in compromised bony sites: a pilot study. *J Oral Maxillofac Surg* 2015; 73: 630-640.
 - 11) Passaretti A, Petroni G, Miracolo G, Savoia V, Perpetuini A, Cicconetti A. Metal free, full arch, fixed prosthesis for edentulous mandible rehabilitation on four implants. *J Prosthodont Res* 2018; 62: 264-267.
 - 12) Vallittu PK. Compositional and weave pattern analyses of glass fibers in dental polymer fiber composites. *J Prosthodont* 1998; 7: 170-176.
 - 13) Sfondrini MF, Massironi S, Pieraccini G, Scribante A, Vallittu PK, Lassila LV, *et al.* Flexural strengths of conventional and nanofilled fiber-reinforced composites: a three-point bending test. *Dent Traumatol* 2014; 30: 32-35.
 - 14) Dyer SR, Lassila LV, Jokinen M, Vallittu PK. Effect of fiber position and orientation on fracture load of fiber-reinforced composite. *Dent Mater* 2004; 20: 947-955.
 - 15) Lassila L, Garoushi S, Vallittu PK, Säilynoja E. Mechanical properties of fiber reinforced restorative composite with two distinguished fiber length distribution. *J Mech Behav Biomed Mater* 2016; 60: 331-338.
 - 16) Lastumäki TM, Lassila LV, Vallittu PK. The semi-interpenetrating polymer network matrix of fiber-reinforced composite and its effect on the surface adhesive properties. *J Mater Sci Mater Med* 2003; 14: 803-809.
 - 17) Khan AA, Al-Kheraif AA, Al-Shehri AM, Säilynoja E, Vallittu PK. Polymer matrix of fiber-reinforced composites: Changes in the semi-interpenetrating polymer network during the shelf life. *J Mech Behav Biomed Mater* 2018; 78: 414-419.
 - 18) Iglesias JG, González-Benito J, Aznar AJ, Bravo J, Baselga J. Effect of glass fiber surface treatments on mechanical strength of epoxy based composite materials. *J Colloid Interface Sci* 2002; 250: 251-260.
 - 19) Day M, Deitzel JM, Gillespie Jr JW, Schweiger S. Influence of sizing formulations on glass/epoxy interphase properties. *Composites Part A* 2014; 63: 59-67.
 - 20) Mahmood H, Tripathi M, Pugno N, Pegoretti A. Enhancement of interfacial adhesion in glass fiber/epoxy composites by electrophoretic deposition of graphene oxide on glass fibers. *Compos Sci Technol* 2016; 126: 149-157.
 - 21) ISO 1172: 1996. Textile-glass-reinforced plastics. Prepregs, moulding compounds and laminates. Determination of the textile glass and mineral-filler content. Calcination methods, 2nd ed, International Organization for Standardization, Geneva, 1996.
 - 22) Jokstad A, Gökçe M, Hjortsjö C. A systematic review of the scientific documentation of fixed partial dentures made from fiber-reinforced polymer to replace missing teeth. *Int J Prosthodont* 2005; 18: 489-496.
 - 23) Bae JM, Kim KN, Hattori M, Hasegawa K, Yoshinari M, Kawada E, *et al.* The flexural properties of fiber-reinforced composite with light-polymerized polymer matrix. *Int J Prosthodont* 2001; 14: 33-39.