

Flexural Properties between Domestic and Dental Glass Fibers Reinforced Polymethylmethacrylate

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Abstract

Objectives: The aim of this study was to examine the flexural properties of PMMA reinforced with domestic glass fibers compared to PMMA reinforced with dental glass fibers. Thirty-two PMMA bar specimens size 10x64x3.2 mm³ (ISO 20795-1) were divided equally into 4 groups (eight specimens for each) according to the types of fibers: 1) no fibers 2) dental fibers 3) silane impregnated domestic glass fibers 4) non-silane impregnated domestic glass fibers. All specimens were subjected to 2,000 cycles of thermocycling. Flexural strength was determined using a three-point loading test set up at a cross-head speed of 5 mm/minute by Universal Testing Machine Model 5566. Scanning electron microscopy was used to examine the microstructure of the cracked surface at 200X and 1000X. The results were analyzed by One-way ANOVA and Tukey HSD ($\alpha=0.05$).

Results: The silane impregnated domestic glass fibers demonstrated the highest flexural strength compared to the others while the non-impregnated domestic glass fibers had the lowest flexural strength.

Conclusions: Reinforcing PMMA with silane impregnated domestic glass fibers could improve its flexural strength.

Keywords: flexural properties, glass fibers, polymethylmethacrylate, silane coupling agent

Introduction

Dentures have largely been used for treating edentulous patients. The most common material for denture base fabrication is polymethylmethacrylate (PMMA) which offers strength, stability, accuracy, pleasing esthetics, and a reasonable cost compared to other materials.^(1,2) Despite these favorable properties, the fracture has frequently been found due to tensile force and compression force from biting and dropping of dentures.⁽³⁾

In order to eliminate the fracture problem and improve its mechanical properties, the denture base has

been reinforced with several materials. Not only can metal wire be added as a strengthener⁽⁴⁾ but various types of fiber can also be used to strengthen PMMA such as glass fiber, aramid fiber, car-bon fiber, ultra-high modulus polyethylene, nylon, and rigid rod polymer filler.⁽⁵⁻⁷⁾ One of the most common materials is glass fiber^(7,8) which is composed of silica (SiO₂) in the form of polymer. In this form, glass fibers exhibit a structure of triangular pyramid, known as tetrahedron, with silicon atoms in the center surrounded by oxygen atoms

at four corners which orientations such as unidirectional, bi-directional, and random. It was found that the direction of the fibers could influence the strength of the reinforced materials; that is unidirectional fibers provide greater strength than multidirectional ones, and fibers that orientate along the long axis perpendicular to the acting force can also increase the strength of the materials.^(9,10) Likewise, the location of the fibers reinforcement also has an effect on the strength. Positioning on the tension side, the fibers can withstand bending better than those being placed on the compression side.⁽²⁾

Another important factor affecting the strength of the denture base is the bonding of fibers to the base of the prosthesis. Previous studies suggested that pre-impregnated E-Glass fibers provide better adhesion to the denture base polymer and have a higher resistance to fracture compared to high modulus polyethylene fibers that have strength but provide low adhesion to dental polymers.⁽²⁾ Apart from pre-impregnation, a process of silanization can also increase the adhesion of glass fibers. In a study conducted by Vallitu⁽¹¹⁾, PMMA was reinforced with silanized E-glass fibers, findings from a scanning electron microscope showed that the surface between the glass fibers and the polymethyl methacrylate material adhered evenly. Although no significant difference was found between pre-silanized E-glass fibers and non-treated E-glass fibers⁽¹¹⁾, some studies discovered that fibers that were impregnated with silane coupling agent had better flexural strength than those untreated.^(7,12)

Silane coupling agent is used to increase the bond strength between the composite fillers and the polymer network. The chemical structure of the silane coupling is R- $(\text{CH}_2)_n\text{-Si-X}_3$. This structural formula consists of two chemical reaction groups: hydrolysable group and organic functional group. The hydrolysable group (group X) is the part that reacts chemically with inorganic compounds. When the hydrolysis occurs, it forms a silanol group (Si-OH), which subsequently reacts with another silanol group on the silica surface creating siloxane linkages known as chemical bonds that are stable. On the other hand, the organic functional group (R group) reacts chemically with organic compounds found between the two groups, and the silane coupling agent has the ability to adhere to silica-based materials such as glass and porcelain.⁽¹³⁾

In dental clinics, the use of silane coupling agents is common, especially the organic silane group called γ - methacryloxypropyl-trimethoxysilane (γ -MPS).⁽¹⁴⁾ The MPS is generally applied as a pre-hydrolyzed agent in a solvent that contains ethanol and water combined with the silane coupling agent in the amount of 1%-5% by volume. According to Bayne's study, it was found that using only a small amount of silane can increase flexural strength and water solubility.⁽¹⁵⁾ This is agreeable to a study by Chaijareenont *et al.*⁽¹⁶⁻¹⁷⁾ who found that the tensile strength and abrasion resistance of the presilanized alumina-reinforced polymethyl methacrylate group were greater than those of the untreated alumina-reinforced polymethyl methacrylate group.

Glass fibers used in previous studies^(2,4,9) were dental fibers. Those fibers had gone through a surface preparation process including preimpregnated or presilanized with a coupling agent that affected their adhesion to polymethyl methacrylate. Due to the high price of imported dental fibers, however, domestic industrial glass fibers that are cheaper could serve as a substitute to reinforce the polymethyl methacrylate. The purpose of this study is to compare the flexural properties of polymethyl methacrylates (PMMA) reinforced with domestic glass fibers to PMMA reinforced with commercial dental glass fibers.

Materials and Methods

Thirty-two PMMA bars, size 10x64x3.2 mm³ were fabricated (ISO 20795-1 and ADA specification No.12) and divided into four groups (n=8).

Fibers preparation

For Group 2: Commercial dental fibers were prepared in the size of 8.5x65x2.0 mm³.

For Group 3: Domestic glass fibers (J.N.Transos, Samutsakorn, Thailand) were prepared in the size of 8.5x65x2.0 mm³ by boiling at 100°C for 1 hour before drying at room temperature for 24 hours. After that, silane (Monobond-STTM, IvoclarVivadent, Ontario, Canada) was applied and the fibers were again left to dry for 20 minutes to eliminate excess silane. Lastly, the fibers were then heated at 50°C for 1 hour.

For group 4: Domestic fibers were boiled at 100°C for 1 hour and left to dry at room temperature for 24 hours.

Specimens fabrication

Metal specimen bars in the sizes of $45 \times 65 \times 1.2 \text{ mm}^3$ (A) and $45 \times 65 \times 3.5 \text{ mm}^3$ (B) were fabricated. Mold preparation was done in a two-part mold using Hanau Varsity Flasks (Whip Mix, Louisville, KY). The metal bars A and B were coated with two layers of Vaseline (Unilever, Rotterdam, The Netherlands) and then invested in vacuum-mixed type III dental stone (Elite Model, Zhermack, Badia Polesine, Italy) by mixing powder and water at the ratio of 100g: 50ml in the lower half of the flask. After the stone was set, metal bar A was removed and replaced by metal bar B. At this point, the upper half of the flask was then placed and vacuum-mixed type III dental stone was invested. Once the dental stone was set, the

flasks were opened and the metal patterns were removed creating a Mould space for PMMA specimens. (Figure 1) The surfaces of the rectangular cavities were then sealed with two coats of Cold Mold Seal (PSP Dental, Belvedere, UK). The heat-cured PMMA (Triplex hotTM, Ivoclar Vivadent, Ontario, Canada) with a monomer to polymer ratio of 10ml: 23.4g was measured and mixed to reach the dough stage before being placed in the mold cavity. The flasks were separated with cellophane paper. Trial closure was performed with hydraulic pressure at 70 psi and flash was removed. (Figure 2) For groups 2, 3, and 4, (Figure 3) during the trial pack, different rein-forced fibers were placed in PMMA dough material between the separated flasks as listed in Table 1.

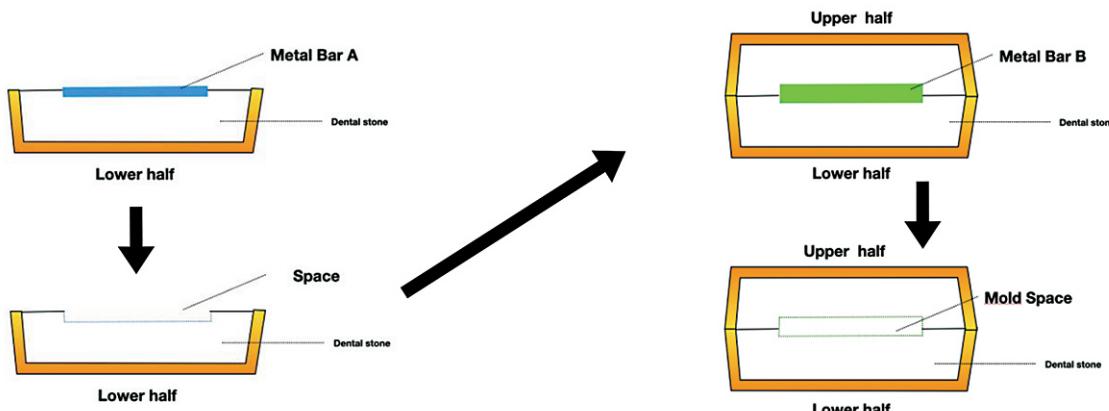


Figure 1: Diagram of mold preparation for test specimen fabrication.

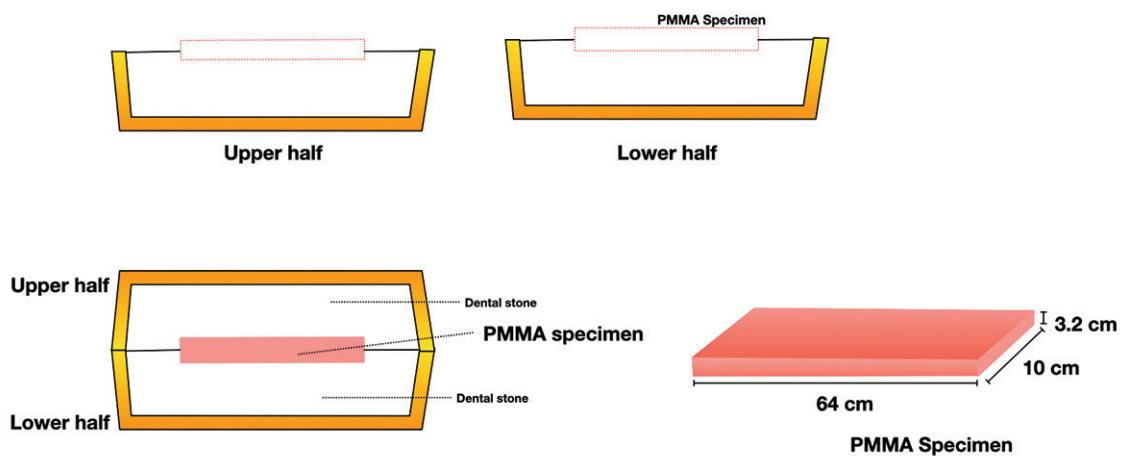


Figure 2: Diagram of test specimen fabrication process for group 1.

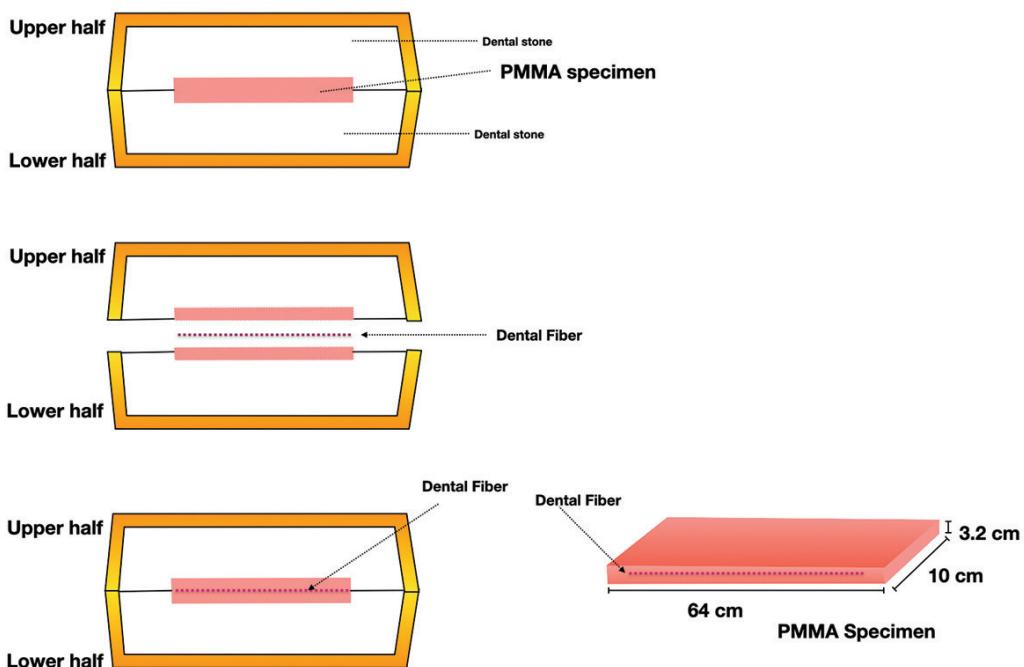


Figure 3: Diagram of test specimen fabrication process for group 2, 3 and 4.

Table 1: Specimen and type of glass fibers.

Group	Reinforced fibers
1	None
2	Commercial dental fibers (Interlig, Angelus, Londrina, PR, Brazil)
3	Silane impregnated domestic glass fibers (J.N.Transos, Samutsakorn, Thailand) (Monobond-S™, IvoclarVivadent, Ontario, Canada)
4	Non-impregnated domestic glass fibers (J.N.Transos, Samutsakorn, Thailand)

After that, the dough was processed under pressure and polymerized using a short curing cycle at 100°C for 1 hour according to the manufacturer's instructions in a thermostatically controlled water bath. Following the cessation of the polymerization cycle, the flask was allowed to cool in the water bath at room temperature for 1 hour before deflasking.

Subsequently, excess acrylic was trimmed and the specimens were laser cut into the size of 10x64x3.2 mm³ before being wet finished with 100, 500, and 1000 grit SiC abrasive papers in a rotational polisher (1 minute per grit, ISO 20795-1). The specimens' dimensions were verified using a digital caliper (Mitutoyo). Thereafter, those specimens were then stored in water at 37°C for 24 hours and thermocycled for 2,000 cycles of alternating 5°C and 55°C water baths with 20-second dwell time.

After the storage, the specimens were tested in a universal testing device (Model 5566, Instron® Co., USA). (Figure 4)

Flexural strength was determined using a three-point loading test set up at a crosshead speed of 5±1 mm/minute. The strength was calculated from the peak failure load and the surface area of PMMA. Following the testing, the specimens were examined at 200X and 1000X in scanning electron microscopy to identify the microstructure of fracture patterns. One-way ANOVA and Tukey HSD were used to compare the mean flexural strength values among the groups (alpha=0.05).

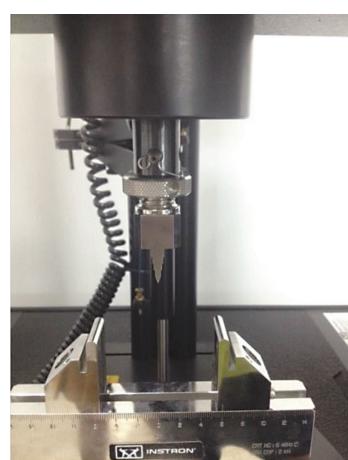


Figure 4: Three-point bending test using Universal testing Machine (Model 5566, Instron® Co., USA).

Result

The results of the flexural bond strength test are presented in Table 2 and Figure 5. The mean values of flexural bond strength of all groups ranged between 83.87 and 105.17 MPa. It was found that silane impregnated domestic glass fibers demonstrated the highest flexural strength values compared to the other groups. The control group and the dental fibers were not significantly different ($p>0.05$) while the lowest flexural strength values belonged to non-impregnated domestic glass fibers.

Table 2: Mean and Standard deviation of flexural strength from each experimental group. Mean (S.D), N=8.

Specimen	Flexural strength \pm S.D (MPa)	Significance ($\alpha=0.05$)
No fiber (control)	84.90 \pm 6.83	b
Dental fiber	83.87 \pm 16.05	b
Domestic fiber W silane	105.17 \pm 9.70	a
Domestic fiber	65.27 \pm 5.45	c

*Values with the same letter were not significantly different at $p>0.05$

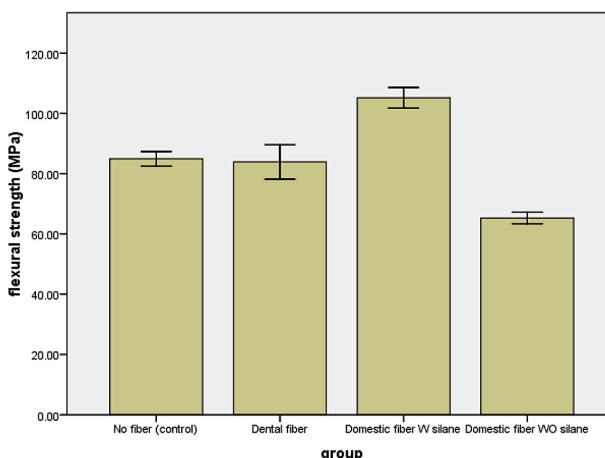


Figure 5: Flexural strength (MPa) of all test groups.

Discussion

The present study investigated the flexural strength properties of domestically-produced glass fiber reinforced PMMA and prefabricated dental glass fiber reinforced PMMA. The result of this study revealed that silane impregnated domestic glass fibers reinforced PMMA demonstrated the highest flexural strength, while non-impregnated domestic glass fibers reinforced PMMA exhibited the lowest flexural strength. In addition, dental

fibers reinforced PMMA demonstrated similar strength to the control group.

This flexural strength test simulated the force of dental prosthesis in oral situations, which includes the pressure, impact, tensile and flexural force exerted on the base of the prosthesis. The result of this study suggested that the incorporation of silane impregnated domestic glass fibers in PMMA can significantly increase its flexural strength.

The present study is in line with previous literature that has generally reported that the reinforcement of PMMA led to significant improvement in the mechanical properties of PMMA. PMMA is a popular material for fabricating dental bases. Regarding previous studies, glass fibers have been the most widely studied material used for strengthening the foundation of prosthetic teeth. Furthermore, there have been several improvements in mechanical properties of the reinforced denture bases such as fracture resistance, impact resistance, and flexural strength.^(4,7,21)

Marei *et al.*⁽²²⁾ stated that stress could be transferred from the PMMA matrix to fibers if the fibers adhere well to the polymer. However, the glass fibers themselves cannot bond directly to PMMA. Many studies thus have investigated the use of silane coupling agent to improve the adhesion between the glass fibers and PMMA.^(7,15,16) Therefore, the result of this study further affirms that the PMMA denture bases can be strengthened with silane impregnated glass fibers reinforced PMMA.

Domestic glass fibers-reinforced polymethyl methacrylate that was not pre-treated with silane coupling agent showed significantly lower flexural strength than the other groups. After carrying out the SEM evaluation of the surface, porosity between the glass fibers and polymethyl methacrylate was witnessed (Figure 6D and 7G), which demonstrated that there was no adhesion between these two materials. This finding agrees with previous studies^(16,17) and again confirms that silane pre treatment of dental fibers can improve the adhesion of fibers to the polymer and consequently increase the flexural strength.

As for commercial dental fibers, they showed slightly lower flexural strength than the control group, although statistically insignificant. This was different from several previous studies^(2,8,10), which indicated that commercial dental fibers provided significantly higher flexural strength than non-reinforced polymethylmethacrylate.

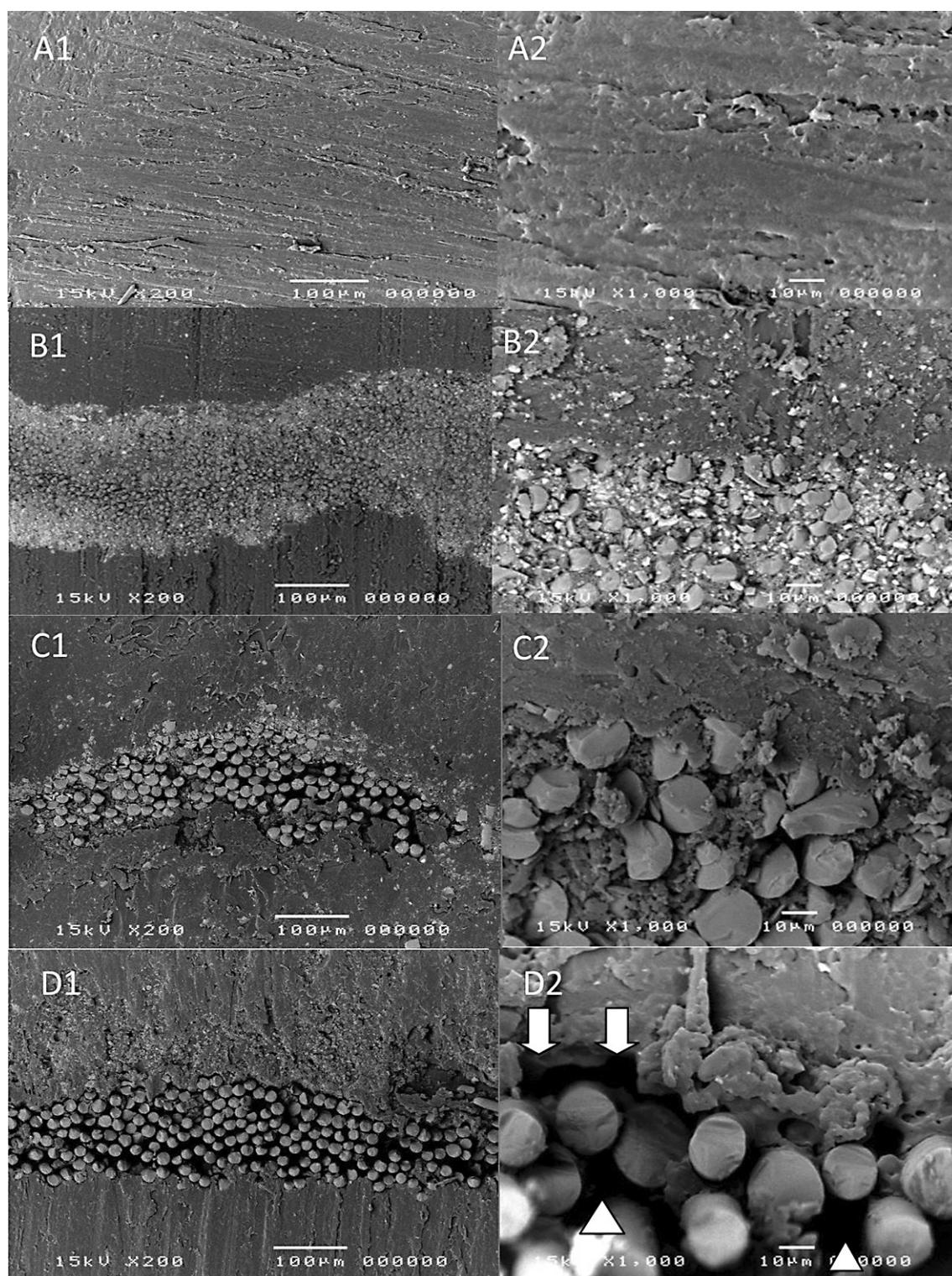


Figure 6: Scanning electron micrographic images of surfaces before 3-point bending test A-D show the cutting surfaces. (A1, A2 PMMA matrix was without glass fibers; B1, B2 PMMA matrix was with dental fibers; C1, C2 PMMA matrix was with silanized domestic fibers; D1, D2 PMMA matrix was with non-silanized domestic fibers; D2 gaps were present both between the domestic fibers and PMMA (arrows) and the domestic fibers and the domestic fibers (arrow heads).

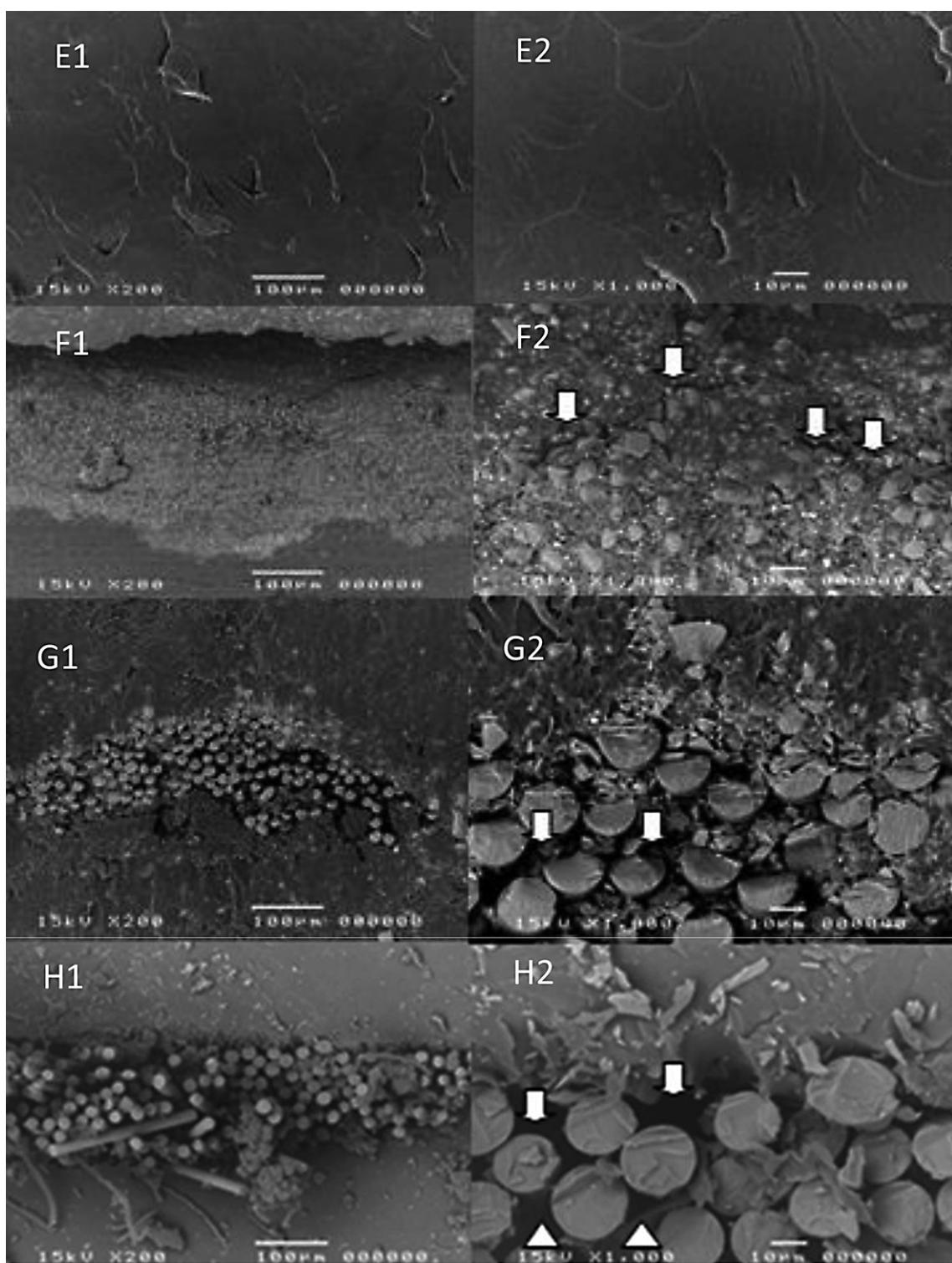


Figure 7: Scanning electron micrographic images of surfaces after 3-point bending test. E-H show the fracture surfaces (E1, E2 PMMA matrix was without glass fibers; F1, F2 PMMA matrix was with dental fibers; F2 Cracked lines were present in dental fibers (arrows); G1, G2 PMMA matrix was with silanized domestic fibers; G2 gaps were present between the domestic fibers and silane (arrows); H1, H2 PMMA matrix was with non-silanized domestic fibers; H2 gaps were present both between the domestic fibers and PMMA (arrows) and the domestic fibers and the domestic fibers. (arrow heads).

This contradiction in the findings could be a result of the different kind of dental fibers used in this experiment. While continuous unidirectional E-glass fibers (StickTech Ltd, Turku, Finland) whose surfaces were pre-treated with light-polymerizing resin were mostly used in previous studies, the materials used in this study were braided dental glass fibers impregnated with light-cured composite resin (Interlig, Brazil). According to Narva⁽²⁾, the alignment of the fibers could impact the tensile strength of the material. It was suggested that the unidirectional arrangement of glass fibers yielded higher cross-sectional strength than woven glass fibers. Since the dental fibers in group 2 of this study were braided glass fibers, this might be a reason for the lower flexural strength.

Conclusions

Within the limitation of this study, it is concluded that silane impregnated domestic glass fibers could increase the flexural strength of PMMA. Additionally, the use of domestic glass fibers reinforced with the silane coupling agent was feasible and cost-effective.

Although domestically produced glass fibers treated with silane coupling agent were proven to increase the flexural strength of the dental base material in this study, further clinical studies should be undertaken before implementing this material for clinical practices.

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