The effects of polymerization methods and fiber types on the mechanical behavior of fiber-reinforced interim restorations

Nan-Chieh Huang, DDS, MDSc,[a] Marco C. Bottino, DDS, MS, PhD,[b] and Tien-Min G. Chu, DDS, PhD[c]

[a]Resident, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN.

[b]Assistant Professor, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN.

[c]Associate Professor, Department of Restorative Dentistry, Indiana University School of Dentistry, Indianapolis, IN.

Corresponding author:

Dr Tien-Min G. Chu

1121 W. Michigan St. Rm. DS112

Indianapolis, IN. 46202

E-mail: tgchu@iu.edu

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[Abstract]

Statement of Problem. Glass fibers have been used for decades to increase fracture resistance in interim restorations. However, poor polymerization between fibers and composite resins can cause debonding and result in failure.

Purpose. The purpose of this study was to investigate the effects of different polymerization methods as well as fiber types on the mechanical behavior of fiber-reinforced interim restorations.

Material and Methods. Specimens, fabricated in various forms, were divided into five groups; one control group and four experimental groups (n=15) based on the type of glass fiber (strip or mesh) and polymerization methods (one-step or two-step) with 15 specimens/group. First, a 0.2 mm thick fiber layer was fabricated using different polymerization methods, and a 1.8 mm composite layer was added on the top to make a bar-shaped sample, followed by a final polymerization. Specimens were tested for flexural strength and flexural modulus. The failure mode of specimens was observed by scanning electron microscopy.

Results. Both fiber types showed significant variations in the flexural strength of test specimens ($F=469.48$, $P<0.05$), but the two polymerization methods did not significantly differ in flexural strength ($F=0.05$, $P=0.82$). Moreover, the interaction between these two variables was not significant ($F=1.73$, $P=0.19$). With respect to the flexural modulus of test specimens, both fiber types and polymerization methods had a significant effect ($F=9.71$, $P<0.05$ for fiber types and $F=12.17$, $P<0.05$ for polymerization methods). However, the interaction between these two variables was not significant ($F=0.40$, $P=0.53$).

Conclusions. Strip fibers showed better mechanical behavior than mesh fibers and should be considered to reinforce interim restorations. However, the choice of polymerization method is not likely to impact reinforcement due to similar effects on the strength and failure mode of fiber-reinforced composites.

Clinical Implications: The type of glass fiber significantly influences the strength of composite resins and therefore should be chosen carefully by clinicians. However, flexibility can be exercised in the preference for polymerization methods.

[Introduction]

Interim restorations are widely used for esthetic and functional purposes in dental clinics. Various materials including polymethyl methacrylate (PMMA), polyethyl methacrylate (PEMA), bis-acryl composite and epimine resin are used to fabricate interim restorations.[1] To increase the strength of these interim restoration materials, they have been reinforced using additional materials such as metal wires, lingual cast metals, carbon fibers, polypropylene fibers, polyethylene fibers and glass fibers.[2]

The effectiveness of fiber reinforcement is known to depend on many variables of the fiber including the quantity, length, form, orientation, adhesion of fibers to the polymer matrix, and impregnation of fibers within the resin.[3-9] A systemic review considered the delamination, wear and debonding of the veneer material to be the main reasons for the failure of fiber-reinforced resin bonded fixed partial dentures.[10] In addition, poor adhesion between the veneer material and fibers was suggested to be the general reason for debonding.[11] Besides, these problems could be overcome, at least in part, by using preimpregnated fibers for reinforcement.[12]

Different fiber patterns have been suggested for various restoration design reinforcements. Strip fibers were used to reinforce interim PMMA or PEMA restorations.[2, 13] Similarly, mesh fibers have been shown to reinforce denture base materials.[14] These and other studies revealed that both mesh and strip fibers can alter specific interim restoration fracture strength and modulus.[15, 16]

Another parameter in interim restorations is the choice of polymerization methods, which largely depend on the clinicians' preferences. In the one-step method, the dentist places fibers on the patient's teeth right next to the space of the missing tooth. The clinician then uses a matrix to apply a composite resin to build the restoration, followed by polymerization. In the two-step method, the clinician first takes an impression, pours cast and then adapts the fibers on the cast,

followed by polymerization. Such polymerized fibers are then moved to the patient's teeth to continue the restoration as described above.

The one-step method is more advantageous because of high efficiency and less time required. In addition, some authors proposed that the one-step method can decrease the formation of a resin-rich inhibited layer and increase the interfacial adhesion between layers.[17, 18] However, it is difficult to apply intra-oral fiber adaptation because intra-oral moisture also affects the adhesion between materials.[19] Despite a large amount of data in this research area, it is not known how the various polymerization methods affect the mechanical properties of reinforced resin based composites, and how the interaction between different fiber types and polymerization methods affect the composite resin reinforcement. Therefore, the purpose of this study was to investigate the effects of different polymerization methods and fiber types on the mechanical behavior of fiber-reinforced interim restorations. We hypothesized that the two-step polymerization groups will have better mechanical behavior than the one-step polymerization groups, and the mesh fiber may better improve mechanical behavior than the strip fibers.

[Materials and methods]

Materials used in this study are summarized in Table 1. Briefly, 200 μm thick strip fibers were obtained from eFiber (PREAT Corp.). Mesh fibers measuring a thickness of 22 μm were from Perma Mesh (PREAT Corp.). The composite used in the study was FILTEK Z250 (3M ESPE). Test methods were as described by the ISO specification 4049:2009, which stipulates the use of 3-point bending.

Thermogravimetric analysis (TGA) was performed with the SDT-Q600 thermogravimetric

analyzer (TA Instruments, USA) to determine the fiber weight content under a nitrogen atmosphere. Fiber specimens (8 to 10 mg) were heated from 18°C up to 650°C at the rate of 10°C/min with a holding time at 650°C for 30 min.

A control group (n=15) and four experimental groups (n=15/group) were fabricated to represent the effects of two different parameters: type of fiber (strip fibers or mesh) and polymerization method (one-step or two-step group). The two different fibers were cut to 25 mm x 2 mm sizes while maintaining the thickness as provided by the manufacturer. To compensate for differences in thickness between the mesh and strip fibers, the mesh fibers were preimpreganted following the manufacturer's instruction and layered to obtain an eight-layer thick mesh fiber strip.

In the control group (C), the composite resin was packed into customized aluminum molds to fabricate rectangular bar shaped specimens (25mm x 2mm x 2mm) (Fig. 1A). All specimens were light polymerized using a dental curing unit (Demi Plus LED Light Curing System, Kerr, USA) with a wavelength of 450 to 470 nm at 1,100 mw/cm^2 both at the top and bottom of the specimens. Six light polymerizing cycles each lasting 5s were necessary to cover the entire length of the specimen (3 cycles on each side).

In the four experimental groups, all reinforcing fibers were oriented to the bottom of the specimens (Fig. 1B). The one-step groups (S/1: strip fiber/one-step; M/1: mesh fiber/one-step) incorporated the composite resin and light polymerization together. Fibers in the two-step groups (S/2: strip fiber/two-step; M/2: mesh fiber/two-step) were light polymerized for 5s first, and then incorporated into the unpolymerized composite resin and lastly light polymerized together. All light-polymerization procedures were the same as the control group.

After fabrication, all samples were polished with the composite polishing kit (Diacomp Composite Polishing Kit, Brasseler, USA). Before testing, all specimens were stored in distilled water at 37 ± 1°C for 24 h.

Flexural strength and flexural modulus were determined using the three-point bending test at room temperature on a universal testing machine (Sintech Renew 1121, Instron Engineering Corp., Canton, MA, USA). All samples were horizontally positioned 20 mm from the two fixed supports at a crosshead speed of 1 mm/min. The data were recorded with the PC software (Test-Works 3.0 MTS Systems Co., Eden Prairie, MN, USA).

All data were initially analyzed by the Levene's test to verify the normality of distribution followed by analysis of variance (ANOVA). One-way ANOVA and Tukey's post hoc test were used to determine the significance among differences in the flexural strength and flexural modulus between the control and experimental groups. The effect of fiber types (mesh, strip) and polymerization methods (one-step, two-step) on flexural strength and flexural modulus among the experimental groups was also assessed using 2-way ANOVA and Tukey's post hoc test. The significance level of all tests was set at 5% in the SPSS 20.0 software. (IBM, New York, USA).

Following mechanical testing, the failure modes of all samples were analyzed manually. The failure mode was categorized into three groups. In group A, both the fibers and composites were completely fractured into two pieces. In group B, either the fibers or composites were fractured. In group C, neither the fiber nor the composite were fractured. In addition, two specimens were randomly chosen from each group to observe the cross-section by scanning electron microscopy (JEOL 7800F, FESEM).

[Results]

Flexural strength of strip fiber groups was significantly higher than the other groups ($P<0.05$) (Fig. 2). However, there was no significant difference in the flexural strength between the mesh fiber groups and the control group. In addition, the 2-way ANOVA results for flexural strength showed significant difference only among fiber types, but not among the polymerization methods and the 2-way interactions (Table 2).

Flexural modulus of the two-step polymerization groups was significantly lower than the other groups ($P<0.05$) (Fig. 3). Statistical analysis by 2-way ANOVA showed significant differences between the fiber types and polymerization steps but no significant difference between the 2-way interactions (Table 3).

Because additional PMMA and bis-GMA were pre-impregnated on strip fibers, we performed the thermogravimetric analysis (TGA) to precisely verify the fiber content. The results revealed a fiber content of 57.93±1.64 wt% in the strip-type fibers (Fig. 4).

Furthermore, SEM images showed that multiple preimpregnated glass fibers were densely compacted into unidirectional strips (Fig. 5A). Dimension of each fiber in the strip fibers was 16 -17 μm (Fig. 5B). However, the mesh fiber was oriented into the net type and loose connections were observed between the fibers (Fig. 6A). The dimension of each fiber was 5-6 μm (Fig. 6B). Although the manufacturer claimed that the mesh fiber was non-impregnated (Table 1), a thin layer of resin was noticed over the mesh fiber structure (Fig. 6B).

Investigation of the failure modes showed that the control group had complete fractures (Table 4). With fiber reinforcement, the fracture mode changed from complete fracture to partial fracture or non-fracture. In addition, the polymerization methods did not change the failure mode in the same fiber materials. However, differences in the partial fractures between the mesh and

strip fiber groups was significant. For example, partial fractures on the strip fiber group had fracture lines between the fibers and composite with the bottom of the fibers still intact. Furthermore, some partial fractures in the mesh fiber groups were close to complete fractures and were barely connected to the mesh fibers.

SEM images of the fracture strip fibers revealed cohesive failure accompanied by the pullout and bending of the fiber strips, as well as the delamination of the composite resin from the fibers (Fig. 7A). Facture cracks were noticed on the composite resin but were not obvious on the fiber strips. In addition, bonding between the fracture fragment of the composite resin and strip fibers was still intact (Fig. 7B). However, SEM images of the fracture mesh fibers showed a different pattern (Fig. 8A); interfacial failure followed by delamination and fracture crack was noted on both the composite resin and mesh fibers. Cavity on the composite resin was evidence to the pullout of the mesh fiber under force. (Fig. 8B).

[Discussion]

The Z250 composite resin material used in this experiment has been widely tested for its original mechanical properties such as flexural strength and flexural modulus. The values for the mechanical properties we obtained for the control group in the present study are consistent with the range reported previously.[20, 21]

Composite restorations fractured at certain weak areas with focal points of high stress from the masticatory forces or impacts outside the oral cavity. Factors that contribute to high stress concentration initiate the cracks. Fiber reinforcement has been proposed to increase the resistance of resin-based composite materials to fracture especially in high stress-bearing areas.[22] Various fiber materials including carbon fibers,[23] polypropylene fibers,[24, 25] polyethylene fibers[26-

[28] and glass fibers[7, 29, 30] have been tested for this purpose. In a fiber-reinforced composite, the fibers can carry the load and effectively resist the stress on the tensile surface. The SEM images in the present study showed that stress was transferred from strip fibers to the composite resin before failure (Fig. 7A). The fracture line that passed through mesh fibers and composite resin was also evident (Fig. 8A).

The findings of the present study are in agreement with previous studies demonstrating that strip glass fiber improved the mechanical behavior of composite resin.[26, 31] However, mesh fiber reinforcement did not show significant differences when compared to unreinforced specimens in this study. This result was not consistent with previous studies.[16, 32-34] A number of factors could have likely caused these results. First, the new composite material used in the present study has better mechanical behavior therefore the mesh fiber does not provide additional reinforcements. In one previous study, the flexural strength of all specimens was less than 100 MPa.[22] In the present study, however, the flexural strength in the control group was 140.5 MPa. This finding was also consistent with the results reported by another group.[35] Second, the need for preimpregantion of the non-impregnated mesh fibers used in the present study may introduce air bubbles and excess monomers,[36] which are likely to inhibit adhesion between the mesh fiber and the composite. Higher magnification SEM images in the present study (Fig. 7B and 8B) also showed that the incorporation of strip glass fibers and composite resin was better with less porosity. Lastly, differences in the fiber diameter may have also led to differences in the load-carrying capacity between the strip glass fiber and mesh fibers (Fig. 5B and 6B).

Furthermore, several studies have indicated that multidirectional E-glass fiber cannot be used in combination with composites.[37, 38] A previous study pointed out that the direction of glass fibers critically affects fiber-reinforced polymers and suggested that woven fibers do not reinforce the

denture based PMMA.[39] In addition, Krenschel's factor-based determination of the effectiveness of fiber reinforcement showed that woven fiber was less effective than the unidirectional fiber.[40]

Initially, we hypothesized that the two-step polymerization may improve the mechanical behavior than the one-step polymerization. The result of the present study did not validate this hypothesis. However, a previous study showed that the two-step method improved the overall mechanical behavior of reinforced autopolymerized acrylic resins when compared to the one-step method.[41] In a study that determined the effects of different polymerization sequences during the application of two different composites on fiber-reinforced composite, the authors indicated the need for different polymerization sequences in the material combinations.[17] This study found the effects of the fiber types and interaction between the fiber types and polymerization methods to have significant impact but not the effects of the polymerization methods.

Although many studies have suggested that the fiber-reinforced composites can be used as alternate materials for interim or permanent crown fabrication, in clinical applications these materials are not used interchangeably mostly due to layering procedures. The results of this study revealed no significant differences between the different polymerization methods on the flexural strength of the fiber-reinforced composite resin. Furthermore, tooth-mold samples and clinical studies will be needed to evaluate the effect of different polymerization methods on the mechanical behavior of fiber-reinforced composites. Then, a reliable and applicable method can be developed to decrease the possible complications and treatment difficulties in clinics. Lastly, manufacturers are also encouraged to improve their fiber products for better clinical application.

[Conclusions]

Strip fibers showed better mechanical behavior than mesh fibers and may improve the composite resin reinforcement. However, different polymerization methods did not have significant effects on the strength and failure mode of fiber-reinforced composites.

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Material	Brand	Manufacturer	Chemical composition
Composite resin	FILTEK Z250	3M ESPE Dental Products	Matrix: bis-GMA, TEGDMA,EDMAB and UDMA Filler: 75-80 wt%
Pre-impregnated glass fiber	eFiber	PREAT Corporation	Glass fiber (13μm in diameter) 200μm thickness 100mm length Impregnating with bis-GMA and PMMA resin
Non-impregnated glass fiber	Perma Mesh	PREAT Corporation	22μm thickness 50mm*90mm surface area
Bonding agent	ADPER Single Bond 2	3M ESPE Dental Products	bis-GMA, UDMA, EDMAB, DMA 25-35wt% Ethyl alcohol 5-15wt% HEMA 10-20wt% Nanofiller silica

TABLE 1. Summary of the materials used in this study.

TABLE 2. Statistical significance of the flexural strength (MPa) calculated by Analysis of variance (ANOVA)

Source	Df	Sum of Square	Mean Square	F ratio	P-value
Polymerization methods	1	5465712.71	163.98	0.05	0.82
Fiber type	1	1497236.50	1497236.50	469.48	<0.05
Polymerization methods* Fiber type	1	5522.50	5522.50	1.73	0.19

TABLE 3. Statistical significance of the flexural modulus (MPa) calculated by Analysis of variance (ANOVA)

Source	Df	Sum of Square	Mean Square	F ratio	P-value
Polymerization methods	1	20085381.56	20085381.56	12.17	<0.05
Fiber type	1	16022123.13	16022123.13	9.71	<0.05
Polymerization methods* Fiber type	1	658349.56	658349.56	0.40	0.53

TABLE 4. Failure modes of the specimens categorized according to the location and the propagation of fracture line.

	Control	M/1	M/2	S/1	S/2
A: complete fracture	15	6	8		
B: Partial fracture		9	7	3	6
C: Non-fracture				12	9

FIGURE 1. Schematic diagrams of the samples (a. control group; b. experimental group) used in this study.

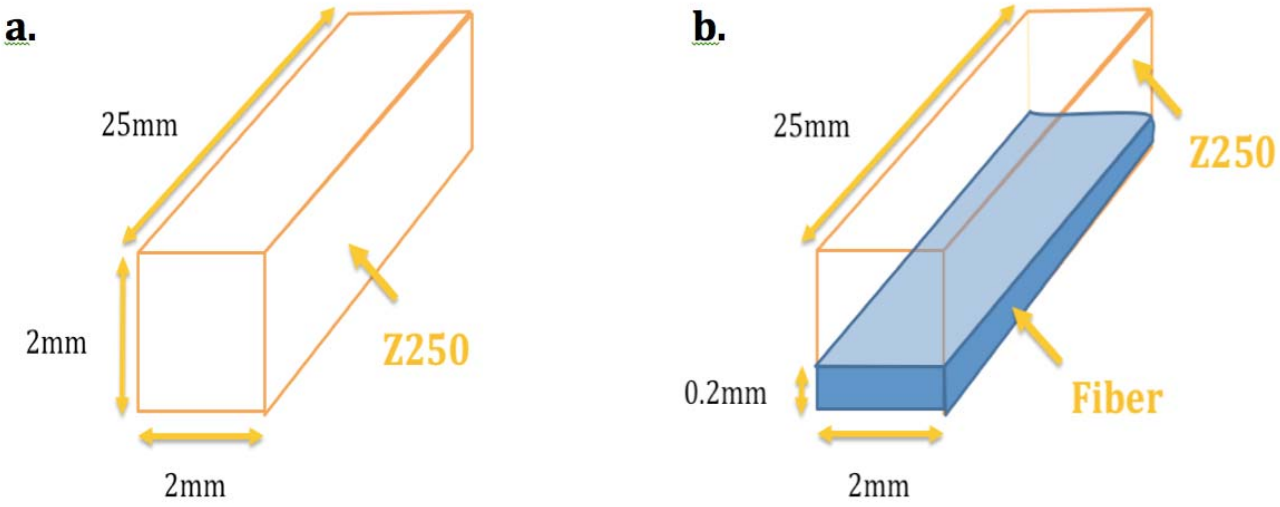


FIGURE 2. Flexural strength of the various tested groups. C – control; M/1 – mesh fiber/one-step, M/2 – mesh fiber/two-step, S/1 – strip fiber/one-step. S/2 – strip fiber/two-step Data represent mean ± standard deviation. Different letters indicate significance at the level of $P<0.05$.

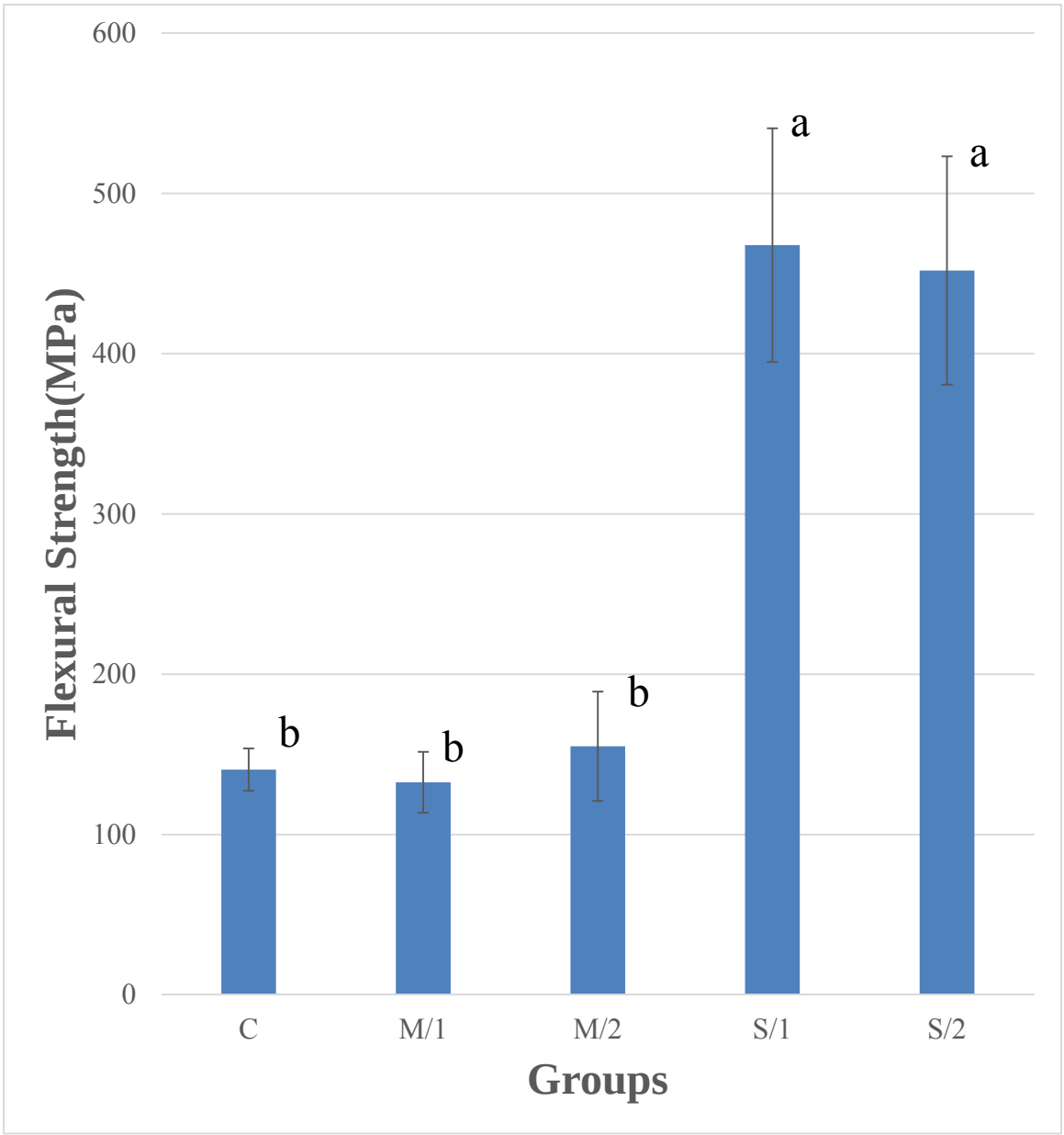


FIGURE 3. Flexural modulus of the various tested groups. C – control; M/1 – mesh fiber/one-step, M/2 – mesh fiber/two-step, S/1 – strip fiber/one-step. S/2 – strip fiber/two-step. Data represent mean ± standard deviation. Different letters indicate significance at the level of $P<0.05$.

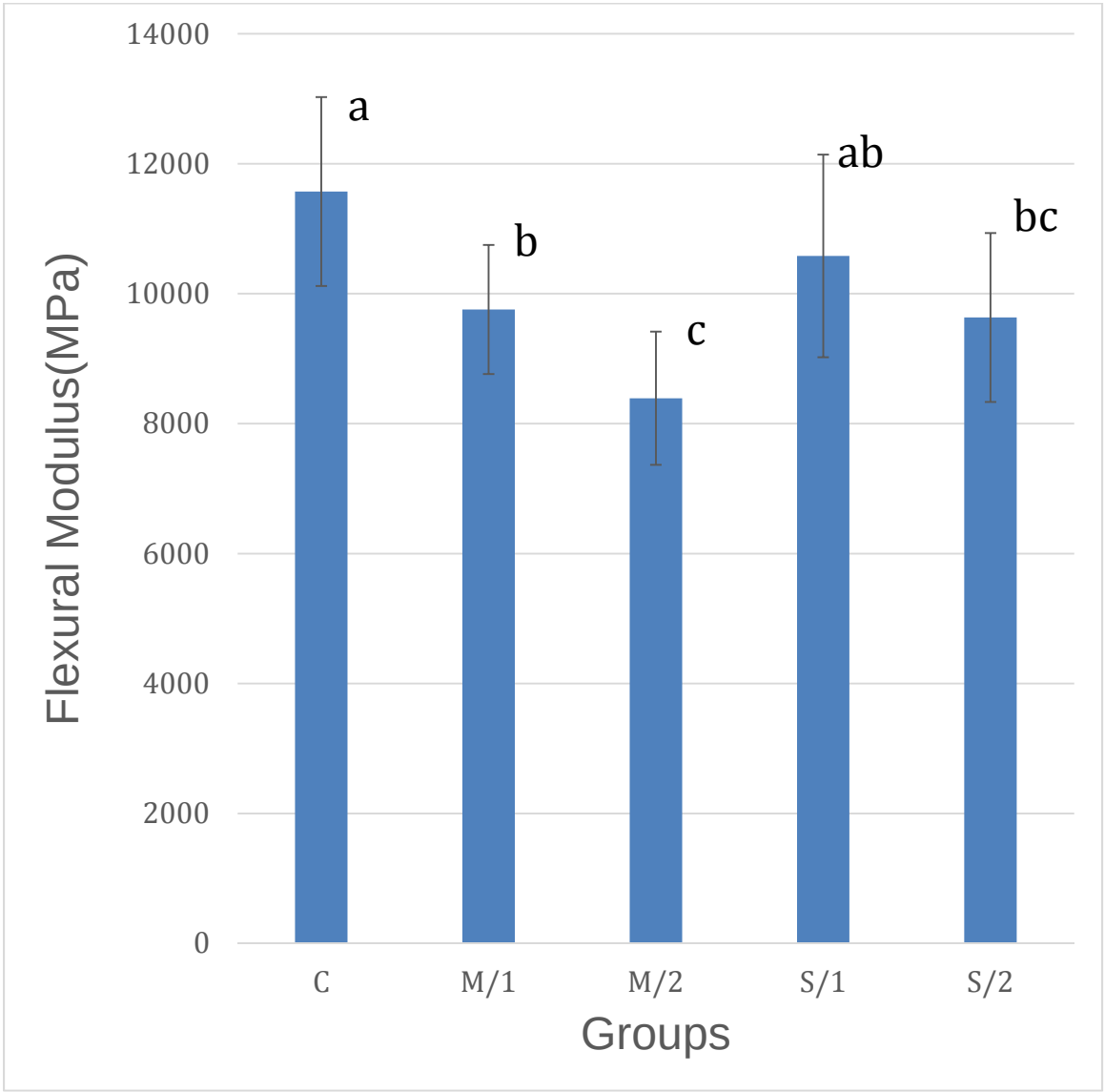


FIGURE 4. Thermogravimetric analysis of the eFiber indicating the amount of fiber in weight (%).

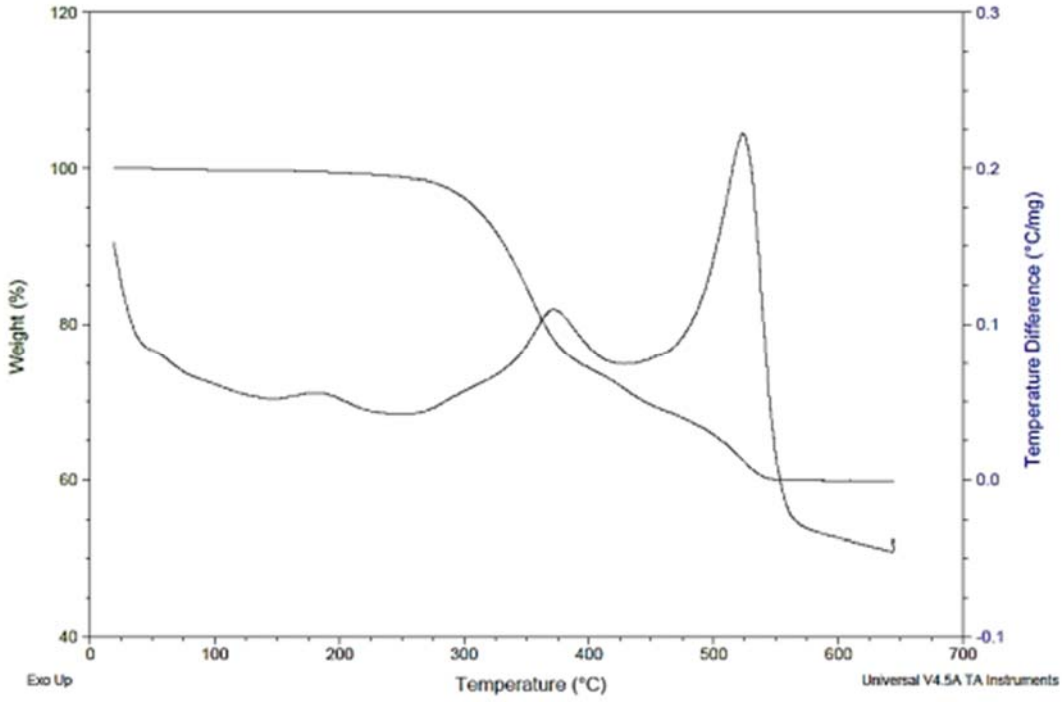
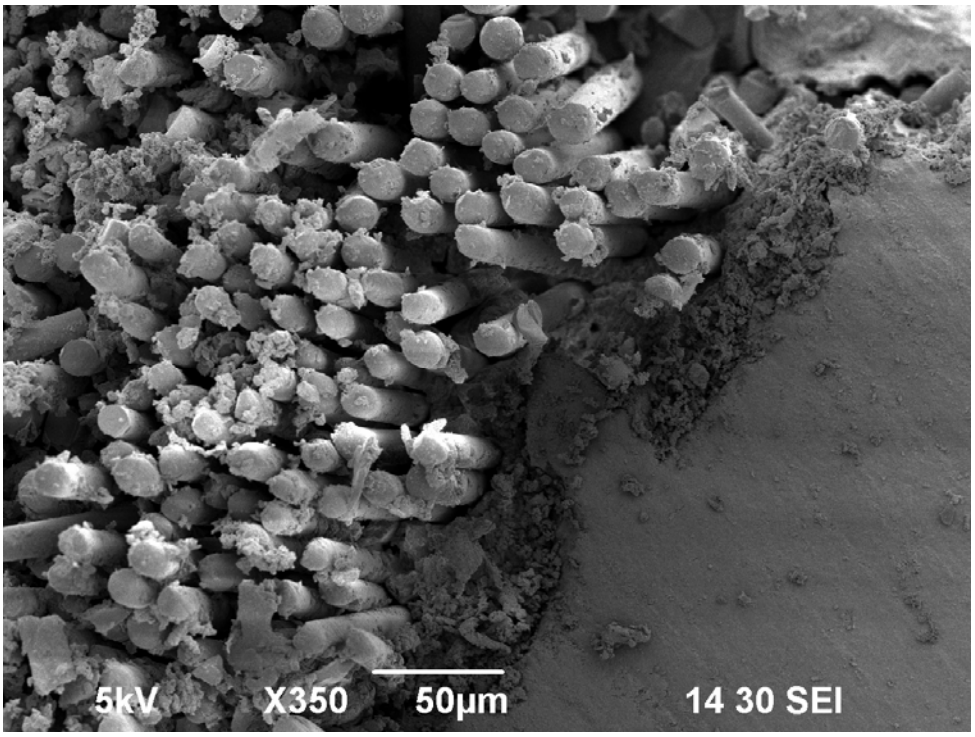


Figure 5. A sample SEM image of eFiber. (A) Fiber arrangement (350X); (B) Fiber after solvent treatment (500X).

A)



B)

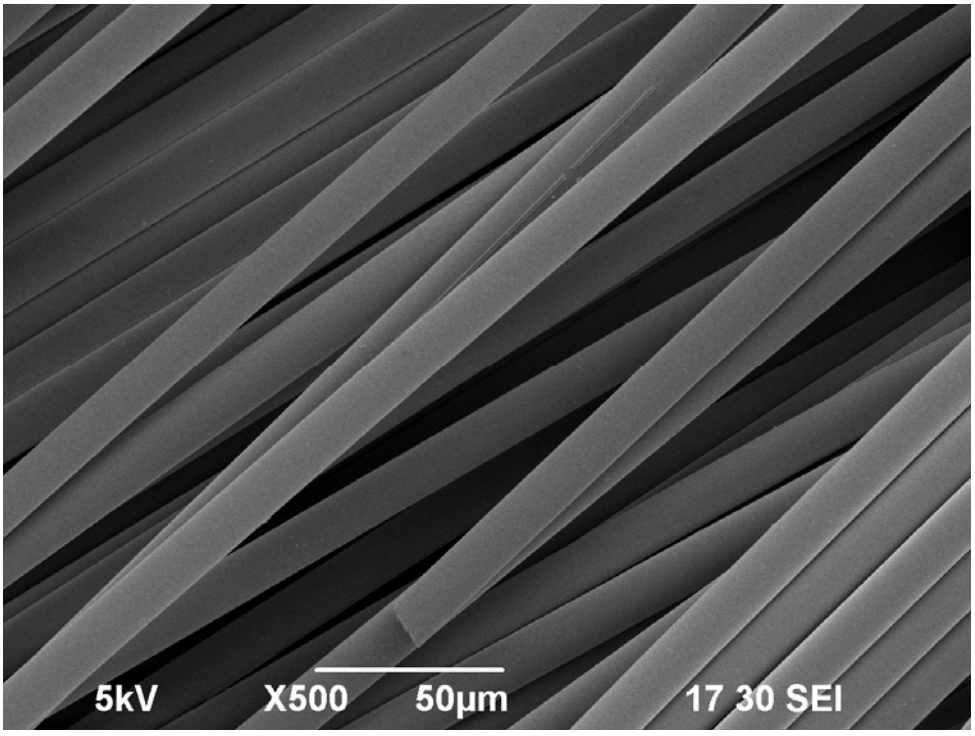
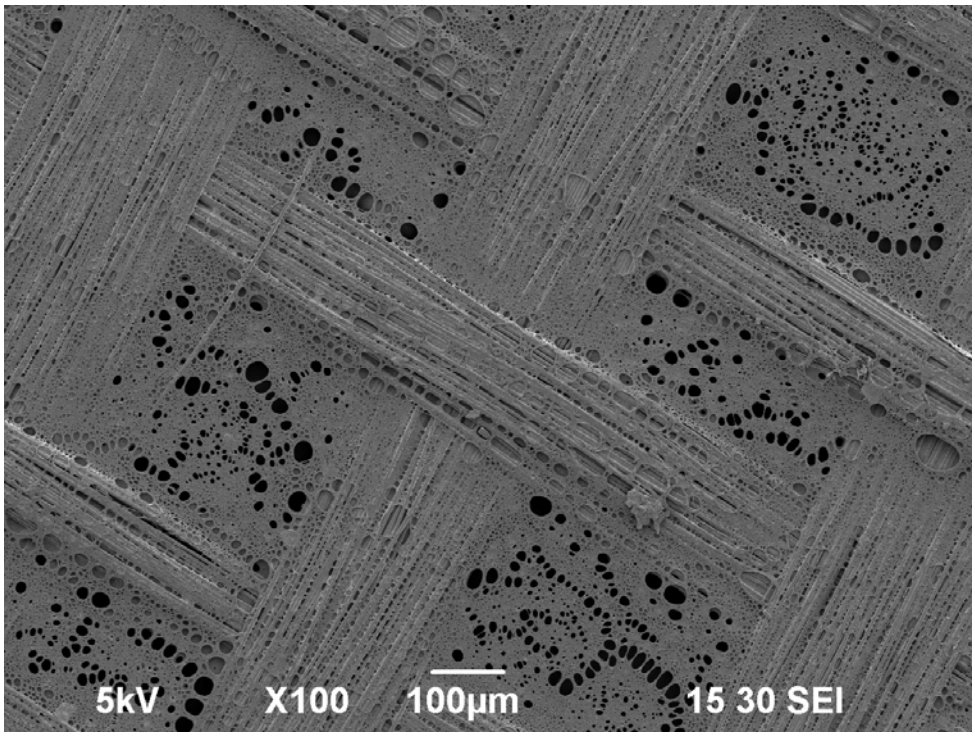


Figure 6. A sample SEM image of Perma Mesh. (A) Fiber arrangement (100X) and (B) fiber characteristics (500X).

A)



B)

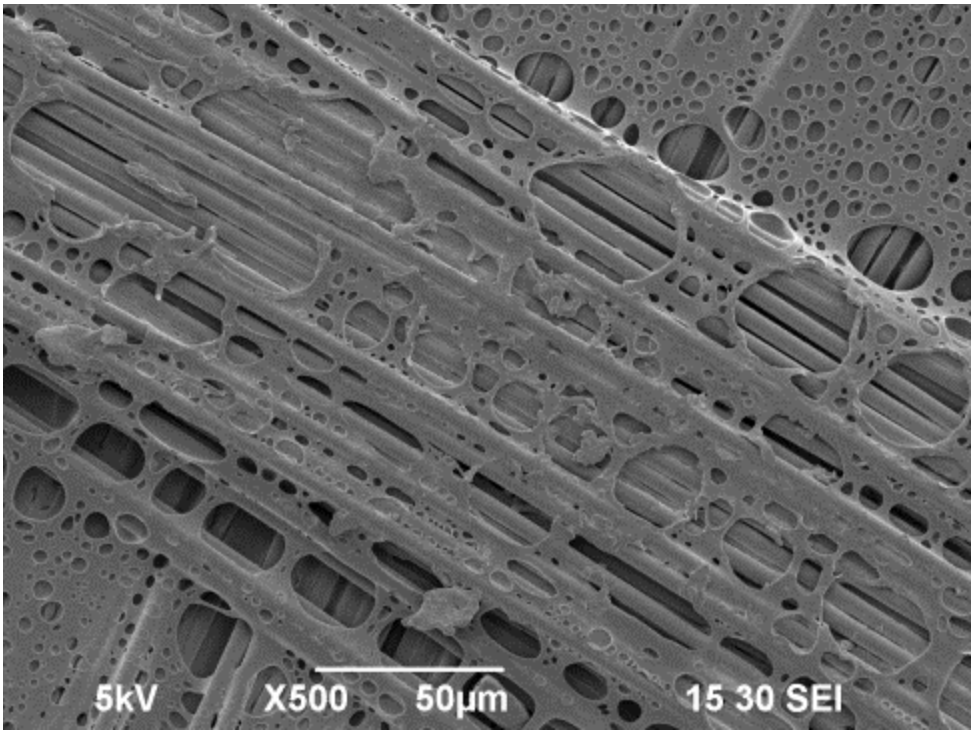
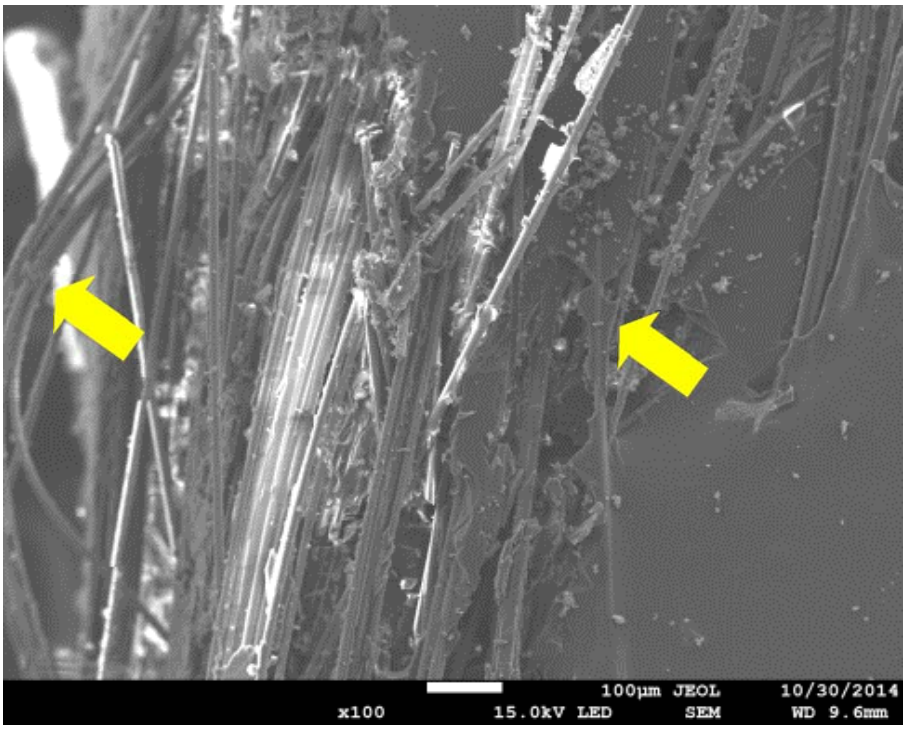


Figure 7. A sample SEM image of fractured eFiber reinforced composite. (A) Both arrows indicate cohesive failure and fiber bending (100X); (B) The circled area indicates intact bonding between eFiber and Z250 composite resin (500X).

A)



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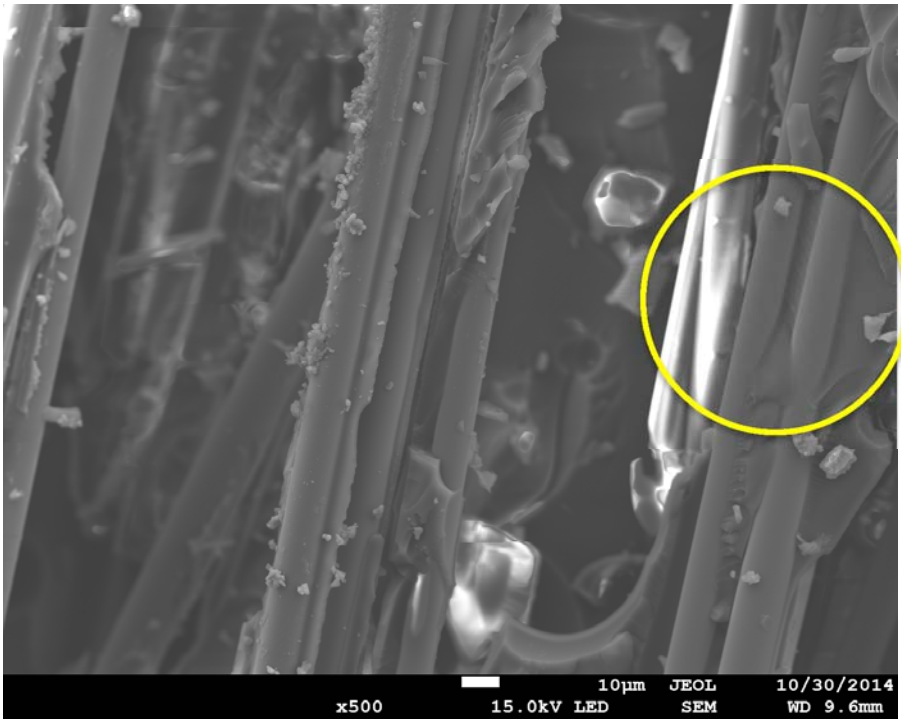
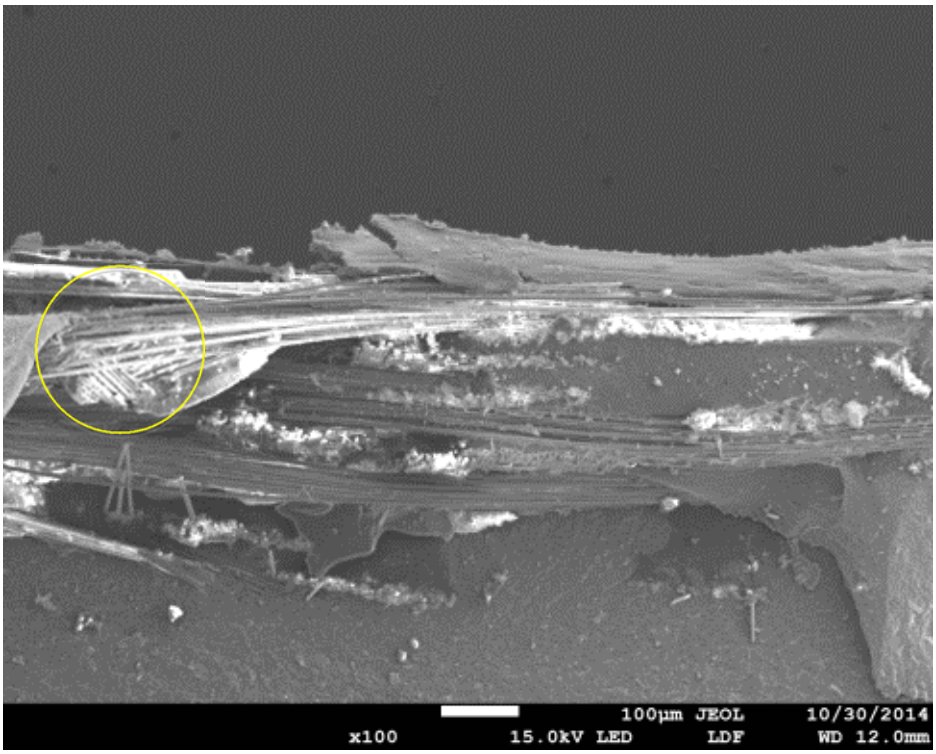


Figure 8. A sample SEM image of a fractured Perma Mesh reinforced composite. (A) Arrow indicates interfacial failure and the circle shows mesh fiber fragments over sample surface (100X); (B) Arrow indicates the space because of fiber pullout (500X).

A)



B)

LEGENDS

Table 1. Summary of the materials used in this study.

Table 2. Statistical significance of the flexural strength (MPa) calculated by Analysis of variance (ANOVA).

Table 3. Statistical significance of the flexural modulus (MPa) calculated by Analysis of variance (ANOVA).

Table 4. Failure modes of the specimens categorized according to the location and the propagation of fracture line.

Figure 1. Schematic diagrams of the samples (a. control group; b. experimental group) used in this study

Figure 2. Flexural strength of the various tested groups. C – control; M/1 – mesh fiber/one-step, M/2 – mesh fiber/two-step, S/1 – strip fiber/one-step. S/2 – strip fiber/two-step. Data represent mean ± standard deviation. Different letters indicate significance at the level of $P<0.05$.

Figure 3. Flexural modulus of the various tested groups. C – control; M/1 – mesh fiber/one-step, M/2 – mesh fiber/two-step, S/1 – strip fiber/one-step. S/2 – strip fiber/two-step. Data represent mean ± standard deviation. Different letters indicate significance at the level of $P<0.05$.

Figure 4. Thermogravimetric analysis of the eFiber indicating the amount of fiber in weight (%).

Figure 5. A sample SEM image of eFiber. (A) Fiber arrangement (350X); (B) Fiber after solvent treatment (500X).

Figure 6. A sample SEM image of Perma Mesh. (A) Fiber arrangement (100X) and (B) fiber characteristics (500X).

Figure 7. A sample SEM image of fractured eFiber reinforced composite. (A) Both arrows indicate cohesive failure and fiber bending (100X); (B) The circled area indicates intact bonding between eFiber and Z250 composite resin (500X).

Figure 8. A sample SEM image of fractured Perma Mesh reinforced composite. (A) Arrow indicates interfacial failure and the circle shows mesh fiber fragments over sample surface (100X); (B) Arrow indicates the space because of fiber pullout (500X).