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Title: Fracture Toughness Enhancement of Fly Ash Based Eco-Core by Glass Fiber Reinforcement

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ABSTRACT

Eco-core material modified by adding glass fibers was developed. Both percent weight and length of the fiber glass were varied. Compression, flexure and fracture properties were measured and compared with base line. Results showed that by adding fiber glass lengths of more than 3mm (1/8") do not improve any of the properties. Fracture toughness expressed in terms of critical stress intensity factor (K_{IC}) can be increased by 2/3 of the base line value by adding 8 % of 3mm long fiber glass. The compromise in compression properties can be restored by increasing the binder content.

INTRODUCTION

Syntactic foams are strong light weight composite materials (density about 0.5 g/cc) and are made by embedding preformed hollow microspheres in a resin matrix. The lightweight hollow microspheres reduce the density of the resin and create a thick mixture that can be applied by hand, sprayed or compression molded. The design advantages of syntactic foams have been recognized for a long time [1] and this type of material has been found to be useful in marine, aerospace, petroleum, and mass transport industries [2-5].

In contrast to the other syntactic foams, the foam developed at North Carolina A&T State University's Composite Materials Research Center (CCMR) is made by sphere-sphere contact of microbubbles coated by a thin layer of high char yield binder. The microrbubbles are Cenosphere, a waste product produced in coal burned thermal power plants. Because this foam has very low binder content and Cenosphere that are exposed to more than 1000°C, when they are produced, the resulting material has excellent fire resistant properties and is called Eco-Core [6-9]. The processing, mechanical and energy absorption properties of eco-core are

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presented in [6-10]. The results show that the base material is brittle and can be damaged by handling abuses. This research focus on improving the eco-core ductility by modifying it by glass fibers.

The material selection, processing, test, and results are presented in this paper.

MATERIALS AND PROCESSING

A class of fly ash known as Cenosphere or Recyclosphere grade SG 300 supplied by Sphere Services Inc, a phenol-formaldehyde resole resin commercially known as Durite SC 1008 supplied by Borden Chemical Co, and chopped E-glass fibers supplied by US composites were chosen. A silane coupling agent, aminoalkyl triethoxysilane, supplied by Aldrich Chemicals was also used.

The fly ash materials were treated to remove contaminants by a dilute HCL acid (pH ~ 4) wash and the heavier than water fraction of the as-received fly ash was separated and removed by settling. The lighter floating fraction material was further washed with water for about 3-4 times and was separated by filtration from the water. It was thoroughly dried at 110°C in a convection oven. Subsequently, the treated fly ash was again treated with a aminoalkyl triethoxysilane coupling agent, as per the instructions from the silane manufacturer. The coupling agent is expected to improve the bonding property. The fly ash after silane treatment was dried in an oven to attain a free-flowing material. The fly ash was then admixed with resole resin diluted with suitable solvents in a low-shear planetary motion mixer to uniformly coat the fly ash particles. The volatile solvents from the fly ash mixture were removed while mixing in a stream of warm air. The coated fly ash mix was subsequently charged into a compression mold of 178 x 178 x 19 mm dimensions and then it was compacted by a laboratory hot press, cured at a pressure of 2.1 MPa and temperature of 163°C for 30 minutes. To achieve reproducibility from sample to sample the void fraction in the foam panels had to be controlled at as low a value as possible. The foam samples were finally post cured in a hot air circulating oven at 163°C for 4 1/2 hours. Specimens extracted from these panels are labeled as baseline.

Chopped glass fibers (12 - 14µm diameter) of various sizes and amounts were predispersed in dry treated fly ash. Glass fiber dispersed cenospheres was admixed with resole resin by a similar process as explained above. The amount of glass fibers in eco-core panels is based on weight fraction of the total compound. Eco-core panels with 3mm (1/8") glass fibers varying in amount from 2 to 8 weight percent (wt.%) were prepared. During dispersion of 6mm (1/4") glass fibers it was observed that amounts in excess of 2 wt.% caused severe flocculation of glass fibers and the dispersion was not uniform. Similar flocculation problems were observed during dispersion of 13mm (1/2") glass fiber in amounts excess of 1 wt. %. Due to this practical limitation, only one eco-core panel with 2 wt.% of 6mm (1/4") glass fiber and two eco-core panels with 1/2 and 1 wt.% of 13mm (1/2") glass fibers were prepared. Enough panels of size 178mm x 178mm (7" x 7") of different material system were made and used for testing.

TESTING

Three types of tests were conducted, namely, compression, flexure and fracture. The compression test was conducted to measure the compression strength and failure modes. The flexure test was used to measure the bending strength of the material. The fracture test was conducted using single edge notched beam (SENB) specimen to measure the fracture toughness and to assess the fracture toughness and modes. Figure 1 shows the specimen layout on a 178mm x 178mm x 19mm panel. This panel size is sufficient to extract four compression samples of diameter 29.2 mm, three flexural specimens of 177.8mm x 19.1mm x 9.7mm, and four 3-point bend (SENB) fracture toughness specimens. Density measurements were carried out on the cored specimens used in compression tests. Compression specimens were represented by C's, flexural specimens by F's and the two fracture specimens were represented by TC for the through-the-thickness crack and MC for the mid-plane crack test. These two specimens will measure the average and mid-plane toughness of the material and will also verify the material uniformity within the panel. The specimen configuration and the loading of compression and SENB specimen are shown in figure 2. The dimensions are $h = w = b = 19.05 \text{ mm}$, $a = w/2$, and $S = 4w$. The notching details of MC and TC specimen are shown in figure 3.

Compression Test

The compression tests were performed according to ASTM C365 using an Instron 4204 electromechanical testing machine. The top and bottom face of each cylindrical specimen was coated with graphite fine powder to reduce the friction in the contact area between specimen and platens. The specimen was compressed between two flat platens at a constant displacement rate of 1.27 mm/min while load and displacement were recorded every half second. Compressive stress and strain were calculated as load/area and displacement/initial height, respectively. Figure 4 shows a typical compression stress-strain behavior for four eco-core samples from the same panel.

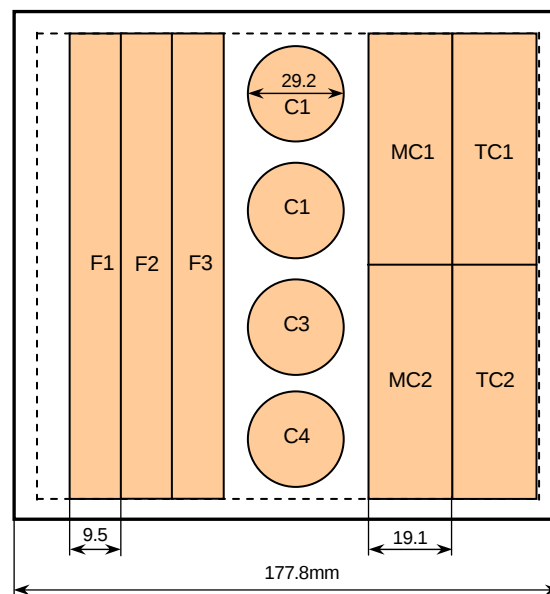


Figure 1. Specimen layout on a panel

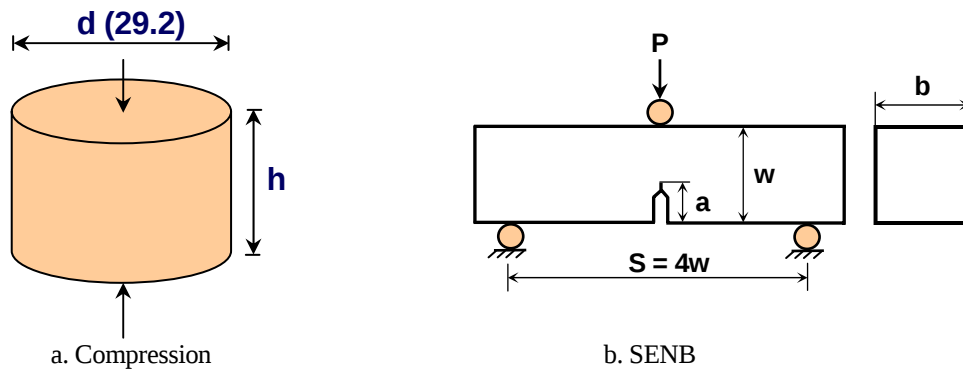


Figure 2. Specimen configurations

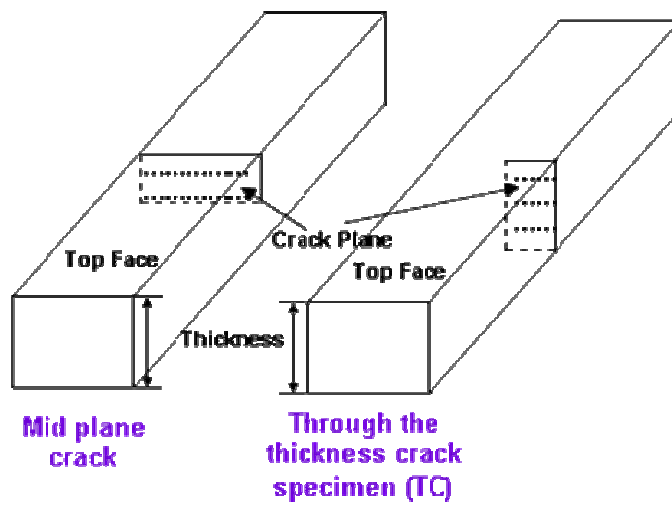


Figure 3. Details of MC and TC crack test specimen

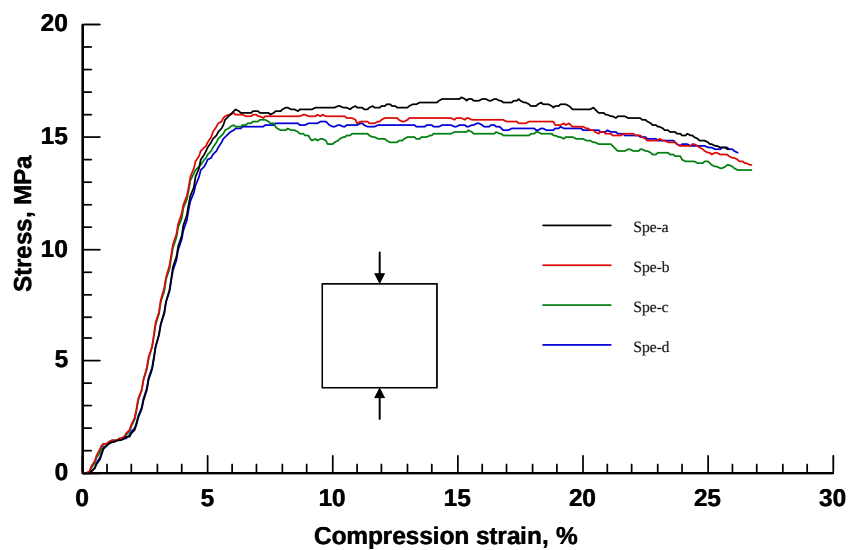


Figure 4. Typical compression stress versus compression strain

TABLE I COMPRESSION AND FLEXURAL PROPERTIES AND FRACTURE TOUGHNESS

Description	Panel ID	Density, g/cc	Compression		Flexural		Fracture Toughness	
			Strength MPa	Modulus GPa	Strength MPa	Modulus GPa	TC MPa-m ^{1/2}	MC MPa-m ^{1/2}
Base line	M02	0.51 (0.02)*	19.1 (1.9)*	0.92 (0.01)*	10.6 (0.9)*	2.69 (0.01)*	0.28 (0.02)*	0.31 (0.02)*
1/8" cgf	2% wt. M10	0.52 (0.01)	17.8 (0.6)	0.82 (0.12)	11.4 (0.4)	2.82 (0.03)	0.31 (0.01)	0.34 (0.01)
	4% wt. M14	0.53 (0.01)	17.0 (0.4)	0.74 (0.02)	11.3 (0.6)	2.79 (0.03)	0.36 (0.01)	0.38 (0.06)
	6% wt. M16	0.53 (0.00)	16.2 (0.8)	0.69 (0.03)	10.2 (0.3)	2.61 (0.05)	0.42 (0.00)	0.44 (0.04)
	8% wt. M04	0.51 (0.01)	12.7 (0.4)	0.61 (0.04)	9.6 (0.1)	2.35 (0.05)	0.50 (0.01)	0.53 (0.05)
1/4" cgf	2% wt. M12	0.53 (0.01)	17.3 (0.9)	0.83 (0.09)	11.1 (0.3)	2.64 (0.03)	0.35 (0.04)	0.35 (0.002)
1/2" cgf	0.5% wt. M22	0.53 (0.00)	18.6 (0.5)	0.86 (0.08)	11.3 (0.1)	2.77 (0.02)	0.29 (0.00)	0.31 (0.00)
	1% wt. M20	0.53 (0.01)	17.7 (1.0)	0.87 (0.09)	11.5 (0.2)	2.87 (0.03)	0.29 (0.01)	0.31 (0.00)
6% wt. 1/8" cgf with 25% binder	M24	0.53 (0.01)	17.1 (0.5)	0.81 (0.04)	11.4 (0.2)	2.71 (0.05)	0.49 (0.07)	0.422 (0.13)

* Standard Deviation

Stress-strain response is almost linear till the maximum stress is reached. The material crushes through the thickness at nearly constant stress till material is completely crushed. The stress becomes constant for further compression and the next layer is crushed. This constant stress-strain response shows the high compressibility and thus higher energy absorbing capability of the material. The core's fracture strain is in excess of 25%. Similar behavior is observed by Gupta, et al. with syntactic foams made with epoxy resin and glass hollow microspheres [3]. Table I summarizes the density, compression strength, and modulus of baseline and glass fiber modified eco-core.

Flexural Test

The flexural tests were performed according to ASTM D-790. The tests were done using a 3-point bend fixture on an MTS hydraulic load frame. Specimens of dimensions 177.8mm x 19.1mm x 9.7mm are used for testing. A support span-to-depth ratio of 16:1 is used. A constant displacement rate of 1.27 mm/min was used while recording load and center deflection every half second. Flexural strength and modulus were calculated and are listed Table I.

Fracture Test

The Fracture toughness tests were performed using single edge notched bend specimen according to ASTM E399. As mentioned already, two types of specimen were selected for the evaluation, through-the-thickness (TC) and mid-plane (MC) crack. Measurement of both through-the-thickness and mid-plane cracked fracture toughness will help identifying any non-uniformity in the material properties. The identical toughness confirms the uniformity of the material and quality of the manufacturing process. The crack starter notch of each sample was machined to a width of 2.3 mm and a depth of about 10.2 mm. A sharp crack was made using a sharp razor blade fixture mounted in a vise. This setup ensured that a sawing motion

against the end of the starter notch would result in a fine crack extending from the center of the starter notch. These cracks were cut to about 1.3 mm beyond the machined notch, to a total crack length to width ratio of 0.45. The tests were conducted using a 3-point bend fixture on an MTS hydraulic load frame at a constant displacement rate of 0.25 mm/min while load and center displacement were recorded every half second. Peak load was used in the following equation to calculate the fracture toughness K_{IC} .

$$K_{IC} = \frac{P}{BW^{1/2}} f\left(\frac{a}{W}\right) \quad (1)$$

$$f\left(\frac{a}{W}\right) = \frac{3\left(\frac{a}{W}\right)^{0.5}}{\left[2\left\{1+2\left(\frac{a}{W}\right)\right\}\left(1-\frac{a}{W}\right)\right]^{0.5}} \left[1.99 - \left(\frac{a}{W}\right)\left(1-\frac{a}{W}\right)\left\{2.15 - 3.93\left(\frac{a}{W}\right) + 2.7\left(\frac{a}{W}\right)^2\right\}\right] \quad (2)$$

Table I summarizes the fracture toughness data for all the eco-core samples. The added fiber increased the material toughness and hence reduces the brittleness of the material. However, the addition of 1/2" glass fibers in amounts of 0.5 wt. % and 1 wt. % did not show much increase in toughness.

RESULTS AND DISCUSSION

Compression Properties

Table I summarizes density, compression strength and modulus, flexural strength and modulus and fracture toughness of baseline and various glass fiber modified eco-core panels. The density ranged from 0.51 g/cm³ for baseline to 0.53 g/cm³ for glass fiber modified specimens. Figure 5 and 6 shows the effect of weight fraction of 1/8" glass fibers on compression strength and compression modulus respectively. It is observed from the figure 5 that the compression strength decreased linearly with increasing wt.% of glass fiber. This may be due to the increased wetting of the resin by glass fiber rather than by the cenospheres. Increase in the amount of glass fiber reduced the quantity of binder available for encapsulating microscopic voids and microbubbles. This phenomenon leads to decrease in compression strength. At 2 wt.%, compression strength of eco-core samples containing 1/4" glass fiber was marginally less than that of 1/8" glass fiber reinforced eco-core samples. It also observed that there is no significant difference in the compression strength of eco-core samples containing 1/2" glass fiber and 1/8" glass fiber. Figure 6 shows that compression modulus is decreased linearly with increasing wt.% of glass fiber. From Table I it is clear that at 2 wt.%, there is no significant difference in the compression modulus of eco-core samples containing 1/4" and 1/8" glass fiber. Compression modulus of eco-core samples containing 0.5 wt.% and 1 wt. % of 1/2" glass fibers is marginally higher than that of the sample containing 2 wt.% of 1/8" glass fibers. In summary, glass fibers longer than 1/8" have no benefit instead they can deteriorate the compression properties.

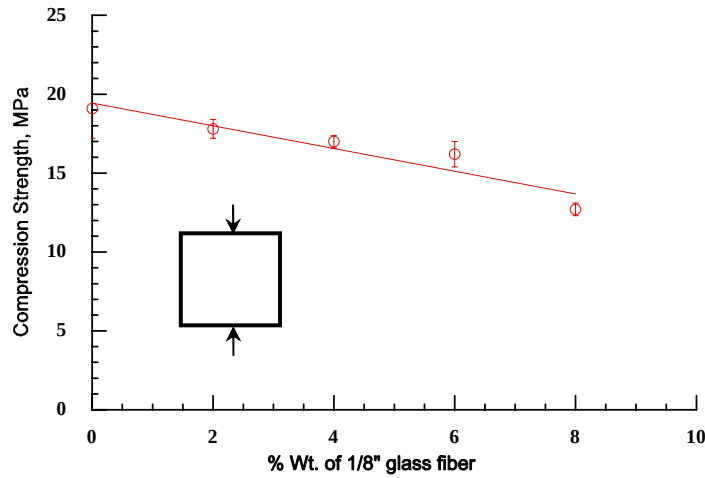


Figure 5. Plot of Compression strength Vs Weight fraction of 1/8 '' glass fiber

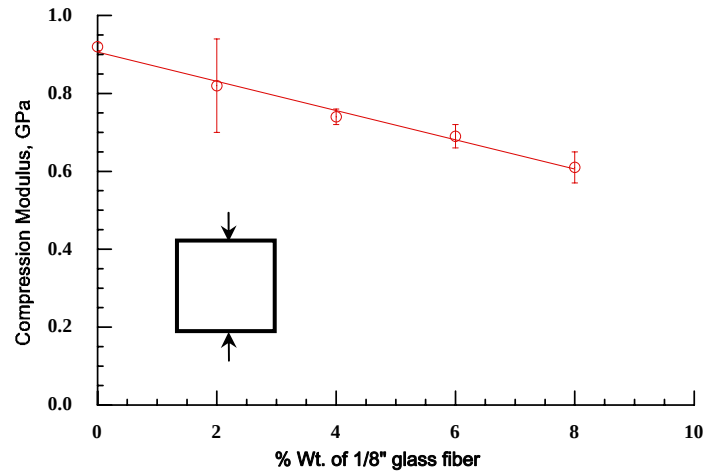


Figure 6. Plot of Compression modulus Vs Weight fraction of 1/8 '' glass fiber

Flexural Properties

The flexural strength and modulus of baseline eco-core and various glass fiber modified eco-core panels are listed in Table I. Figure 7 and 8 shows the effect of weight fraction of 1/8'' glass fibers on flexural strength and flexural modulus respectively. It is observed from the figure 7 that the flexural strength varies (increases) non-linearly with increasing wt.% of glass fiber till 4 wt.% there afterwards it starts decreasing. At 2 wt.%, flexural strength of eco-core samples containing 1/4'' glass fiber and 1/8'' glass fiber are comparable. Also there is no significant difference in the flexural strength of eco-core samples containing 1/2'' glass fiber and 1/8'' glass fiber. Figure 8 shows that flexural modulus is increasing with increasing wt.% of glass fiber till 2 wt.% there afterwards it starts decreasing in a non-linear manner. Flexural modulus of eco-core sample containing 2 wt.% 1/4'' glass fibers is marginally less than that of the sample containing 2 wt.% of 1/8'' glass fibers. It also found that there is no significant difference in the flexural modulus of eco-core samples containing 1/2'' glass fiber and 1/8'' glass fiber.

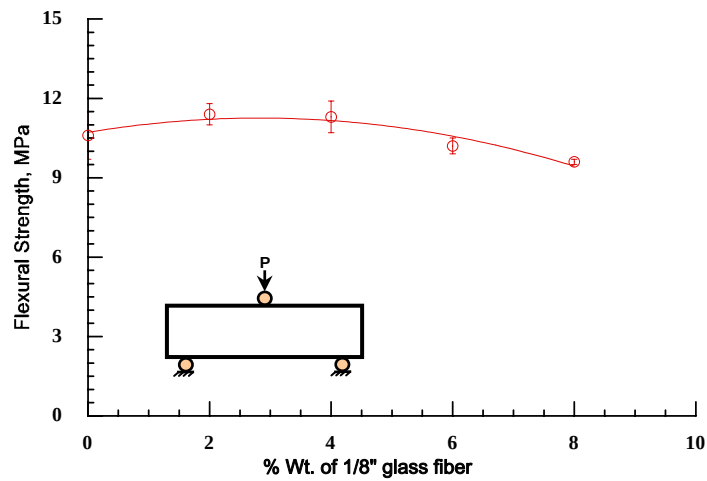


Figure 7. Plot of Flexural strength Vs Weight fraction of 1/8 '' glass fiber

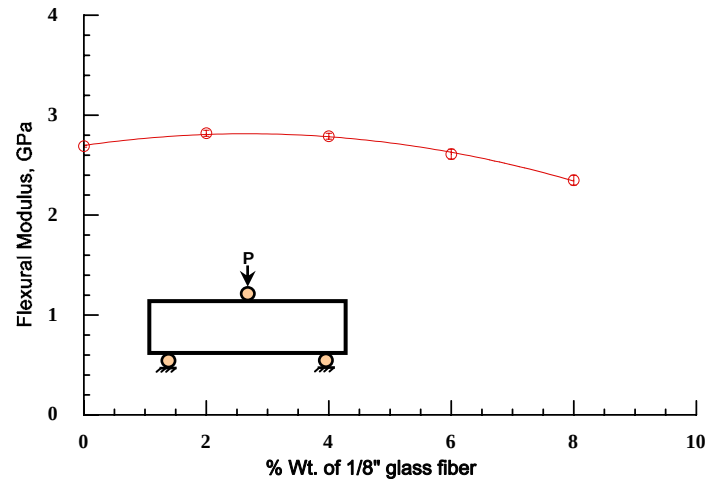


Figure 8. Plot of Flexural modulus Vs Weight fraction of 1/8 '' glass fiber

Fracture Toughness

The fracture toughness results are average of 4 tests. All glass fiber modified eco-core panels showed enhancement of fracture toughness. Among all the eco-core panels, the panel containing 8 wt.% of 1/8'' glass fiber showed very significant improvement (66% increase) in fracture toughness. But this had a penalty of 28% decrease in compression strength. It has shown in reference [10] that the compression strength of the eco-core increases with increase in binder content. In order to maintain the compression strength, the binder content in eco-core panel was increased from 20 to 25 wt.%. It is evident from the Table I that with increased binder content, 66% improvement in fracture toughness is achieved with out compromising in compression strength. Figure 5 shows the effect of weight fraction of 1/8'' glass fibers on fracture toughness. It is observed that the fracture toughness is increasing with increasing wt.% of glass fiber in a non-linear manner. This increased trend in fracture toughness is due to the increased resistance of embedded glass fiber to the crack propagation.

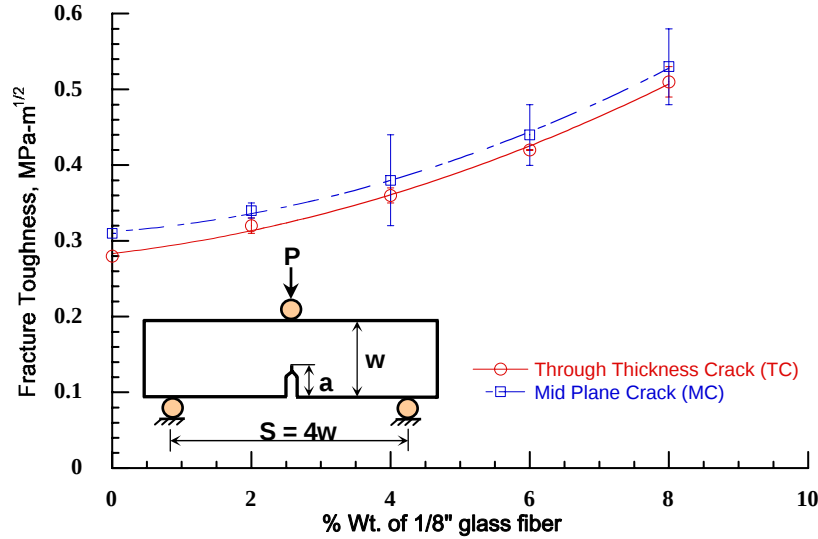


Figure 9. Plot of Fracture Toughness Vs Weight fraction of 1/8 '' glass fiber

At 2 wt.%, no significant difference is seen between the fracture toughness of eco-core samples with 1/8'' glass fiber and 1/4'' glass fiber. It also found that the fracture toughness of eco-core samples containing 0.5 wt.% and 1 wt.% of 1/2'' glass fibers were less than that of 1/8'' and 1/4'' glass fiber reinforced eco-core samples. This reduction in fracture toughness may be due to the longer length glass fibers not uniformly dispersing in the material.

CONCLUSION

Eco-core material modified by adding glass fibers was developed. Both percent weight and length of the fiber glass were varied. Compression, flexure and fracture properties were measured and compared with base line. Results showed that by adding fiber glass lengths of more than 3mm (1/8'') do not improve any of the properties. Fracture toughness expressed in terms of critical stress intensity factor (K_{IC}) can be increased by 2/3 of the base line value by adding 8 % of 3mm long fiber glass. The compromise in compression properties can be restored by increasing the binder content.

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