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Flexural and Fracture Properties of Glass Fiber Reinforced Polyester Polymer Concrete

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Flexural behavior of a polyester polymer concrete was investigated by varying the polymer and fiber contents. The polymer content was varied up to 18% of the total weight of polymer concrete (PC). The chopped glass fibers were 13 mm long and the fiber content was varied up to 6% (by weight of PC). The fine aggregates were well graded with particle size varying from 0.1 to 5 mm and were mainly quartz. The fine aggregates and glass fibers were also pretreated with a coupling agent (γ -methacryloxypropyltrimethoxy silane, γ -MPS) to improve flexural and fracture properties of PC. In general, addition of fibers increased the flexural strength, failure strain (strain at peak stress) and fracture properties but the flexural modulus of PC remained almost unchanged. Addition of 6% fiber content and silane treatment of aggregates and fibers increased the flexural strength of 18% PC to 41.6 MPa (6,040 psi), almost doubling the strength of unreinforced 18% PC system. Crack resistance curves based on stress intensity factor (K_R-curve) have been developed for the fiber reinforced PC systems. A two-parameter relationship was used to predict the complete flexural stress-strain data. There is good agreement between the predicted and measured stress-strain relationships.

<u>Keywords</u>: Coupling agents; esters; fibers (discrete fibers); flexural strength; fracture properties; polymer concrete

2 Vipulanandan and Mebarkia

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INTRODUCTION

Polymer concrete is produced using dry aggregates as filler and polymerizing monomers as binder. The composition of PC is determined by its applications, especially loading stress levels and chemical environment. The high-strength, rapid-setting and corrosive resistance makes polymer composite a potential material for structural repairs, and for new constructions which are regularly exposed to strong alkaline environments (1-4).

Polyester polymer is one of the most popular polymer binders used in PC (5). PC exhibits brittle failure (6,7) and therefore improving its post-peak stress-strain behavior is important. Hence developing better PC systems and also characterizing the flexural strength and fracture properties in terms of constituents are essential in aiding the efficient utilization of PC. In order to improve the post-peak behavior and toughness, glass fibers can be added to the PC matrix. Substantial experience and broader knowledge of the optimal compositions, properties and stress-strain relationships of the fiber reinforced PC are necessary with respect to design, production and quality control. The post-peak behavior and the strain softening stress-strain relationship are essential in evaluating the performance of the material for impact, earthquake and fatigue loading.

In this study, the flexural properties of the glass-fiber-reinforced PC are investigated at room temperature. For the PC systems, a well graded blasting sand was used as the filler and polymer content was varied between 10% and 18% by weight of the PC. The glass fibers were added up to 6% by weight of the PC. Also the role of silane coupling agent on the behavior of PC was studied. Relationships have been developed to represent the flexural stressstrain behavior and fracture resistance curve of PC.

RESEARCH SIGNIFICANCE

Polymer concrete is increasingly used in various applications and hence require better characterization of its flexural and fracture properties. Also methods to improve the performance of a polyester based polymer concrete have been investigated.

EXPERIMENTAL PROGRAM

The constituents used in manufacturing the PC are summarized in Table 1. The viscosity of the unsaturated polyester monomer varied between 40 and 50 poise. Polymerization of unsaturated polyester dissolved in styrene (a mix of 65% unsaturated polyester, 35% styrene) is by free radical copolymerization. Cobalt napthenate (0.3% by weight of resin) was used as the promoter and methyl ethyl ketone peroxide (1.5%) was used as the initiator. The Blasting sand with sub-angular particles had a coefficient of uniformity of 5.8. The sand particles were mainly quartz and had a specific gravity of 2.65. The grain size ranged from 0.1 to 5 mm and was compared to fine aggregate recommended by ASTM C33-85 in Fig. 1. The 13 mm long chopped glass fiber elements have up to about 800 glass strands bonded together. The average diameter of a glass strand was 0.013 mm with a fiber tensile strength of 2,500 MPa (363 ksi), and modulus of 70 GPa (10,160 ksi). The silane coupling agent (γ methacryloxypropyltrimethoxysilane, γ -MPS) was introduced into the PC by pretreatment of glass fibers and aggregates (8). The aggregates and glass fibers were treated by wetting them with 2% aqueous solution of silane coupling agent. The trimethoxy group undergoes hydrolysis in aqueous solution and hydroxyl groups are then available to form oxane bonds to the sand and glass fiber surface. The treated aggregates and fibers were allowed to dry at 100^o C for 24 hours prior to mixing with the resin. PC specimens were compacted in three layers in a teflon lined aluminum mold of dimensions 230 mm x 50 mm x 50 mm. All the flexure and fracture studies were performed on 33 mm thick PC specimens. The PC specimens were first cured at room temperature for a day and at 75° C for an additional day prior to testing (9). A total of 92 beam specimens were tested. The experimental program was divided into four series. The first series included 24 unreinforced beams tested for statistical evaluation of the flexural strength, the second series included 36 beams with and without glass fibers, the third series included 24 beams with silane treated aggregates and fibers and the fourth series included 8 silane treated fiber reinforced notched beams. A diamond saw (2 mm thick) was used to notch the specimens to a maximum depth of 38 mm. During the test the cross head speed of the closed loop servo hydraulic testing machine was maintained constant at 0.05 mm/min. The specimens were tested in four-point bending (in some instances, mentioned on the corresponding plots, three-point bending was used). For the notched beam specimens, the crack mouth opening displacement (CMOD) was measured using knife edges glued to the specimen (10). The deflection of the beams was measured using a LVDT (Linear Variable Differential Transducer) with an accuracy of 2.5×10^{-3} mm (10⁻⁴ inch). For each test, both the load versus loadpoint deflection (for all specimens) and the load versus crack mouth opening displacement (for notched specimens) were monitored continuously using X-Y recorders (Fig. 2).

RESULTS AND DISCUSSION

The average density for the unreinforced systems ranged between 1.96 and 2.15 Mg/m³. The porosity was affected significantly by the workability of the mix and varied considerably within the range of the variables. The

4 Vipulanandan and Mebarkia

subjective measure of the workability was estimated based on a scale of four with four being the best and zero the worst. Subjective workability assessment was based on the mix flowability, compactability, fiber balling and handling ease or difficulty. Of the unreinforced systems, the 14% PC had good workability and low porosity. For this reason 24 beams were tested for statistical evaluation of the flexural strength and modulus.

Statistical Analysis

Factors such as material variability (polymer, aggregates, promoter, initiator), degree of compaction and curing conditions will affect the properties of PC. Hence it is appropriate to quantify the variation in strength of the material in terms of mean, standard deviation and distribution. The flexural strength of 14% PC are plotted on a normal probability scale in Fig. 3. A total of 24 specimens were tested. Linear variation of the data on the probability plot (coefficient of correlation R = 0.98) shows that the data may adequately be represented by a normal distribution. The mean flexural strength was 16.45 MPa (2,390 psi) and the coefficient of variation (C.O.V) was 5%. Figure 4 shows the normal distribution of the flexural modulus (R = 0.97). In this case the mean was 11 GPa (1,600 ksi) and the coefficient of variation was 8.5%. Similar distribution was observed for the failure strain and the mean failure strain was 0.15% and the coefficient of variation was 10%.

Effect of Glass Fibers

Several systems have been considered in this study where the polymer content varied from 10% to 18% and the fiber content varied from 0% to 6% (by weight of PC). Enhancement of the unreinforced PC properties may be achieved by addition of glass fibers, treatment with silane coupling agent or both. The general directions of the improved stress-strain curve of the resulting material are shown in Fig. 5. The improvement may be achieved by increasing the (1) strength, (2) failure strain, (3) modulus, or (4) by improving the post-peak relationship.

The first series of specimens were tested in four-point bending to determine the effect of glass fibers on the flexural properties of PC. The following may be concluded from the test results:

(1) Results from a total of 36 specimens are presented in Fig. 6. Within the range of variables investigated, addition of glass fibers improved the flexural strengths of the 14% and 18% PC systems but decreased the strength of the 10% PC system. In the 10% PC system there is inadequate polymer to bind the aggregates and fibers resulting in reduced strength. The flexural strength varied from about 13 MPa (1,890 psi) for the 10% PC with 6% fiber content to about 33 MPa (4,800 psi) for the 18% PC with 6% fiber content. Addition of 6% glass fiber increased the flexural strength of 18% PC by 80%. Flexural stress-strain relationships for the 18% PC system reinforced with glass fibers up to 6% are shown in Fig. 7.

(2) The flexural modulus of 18% PC system was 11 GPa (1,600 ksi) and remained almost unchanged with the increase in fiber content (Fig. 8). The

standard deviation was about 0.94 GPa (136 ksi). The modulus did not increase due to the addition of glass fibers because of the following factors (a) addition of fibers generally resulted in an increase in void ratio due to reduced workability and (b) only up to 6% of fibers were used and fibers partly replaced the aggregates with similar modulus.

(3) Failure strain of 0.17% for the unreinforced 18% PC was increased to 0.6% with the addition of 6% glass fibers. Adding fibers to the PC produced a bridging effect at the crack tip and hence slows the crack propagation which results in an increase of the failure strain (Fig 7).

Stress-strain Relationship

A two-parameter relation (Eq. (1)) was used in predicting the stressstrain relationship of PC (13). This relationship may be written as

$$Y = \frac{X}{q + (1 - q - p) X + p X^{(q + p)/p}}$$
(1)

Y and X are defined as follows:

$$Y = \frac{\sigma}{\sigma_f} \text{ and } X = \frac{\varepsilon}{\varepsilon_f}$$
(2)

where σ and ε are the stress and strain; σ_f and ε_f are the strength and the failure strain and p and q (p and q vary from 0 to 1) are the material parameters to be determined from experimental data. The parameter q is the ratio of secant modulus at peak stress to initial modulus and so the lower the value of parameter g the greater the nonlinear pre-peak behavior of the PC system. This is also reflected by the decreasing value of parameter q with increase in fiber content, for the 18% PC system. The parameter q was equal to 1 for the unreinforced PC and decreased to 0.76 and 0.32 for PC with 4 and 6 wt% fiber contents respectively. The parameter p controls the post-peak curve and is determined by least square fitting the experimental data points. The steeper the descending stress-strain curve, the smaller the p value (p = 0 for brittle material). Figure 9 shows the normalized stress-strain relationships and the predictions agree very well with the experimental results. As shown in Fig. 9 the post-peak curve of the 6% fiber reinforcement descends more rapidly than the 4% fiber system and only the parameter p quantitatively describes this important observation. The parameter p was 0, 0.26 and 0.03 for the 18 wt% PC with 0, 4 and 6 wt% fiber contents respectively.

Effect of Silane Coupling Agent

Using the silane treated constituents in PC improved the workability for the 10% PC system. This improvement is probably due to the modification of the filler surface which became smoother and therefore increased the flowability of the polymer leading to a better dispersion of the constituents. It has been

6 Vipulanandan and Mebarkia

observed that silane treated fillers provide lower viscosities in filled resins than do untreated fillers (14). The PC specimens with treated aggregates (third series with 24 beams) were tested to determine the enhancement in flexural strength due to the silane treatment. The results (average of 2 specimens tested in threepoint bending) are shown in Fig. 10. The increase in flexural strength are compared to the untreated PC. The maximum increase was 70%. It is noted that the treated unreinforced PC with the lowest polymer content (10%) has a higher strength than the untreated PC with the highest polymer content (18%). As shown in Fig.10, the increase in flexural strength due to silane treatment is very much dependent on the polymer content. The highest strength for the silane treated fiber reinforced system (18% polymer and 6% fiber) was 41.6 MPa (6,040 psi). Silane treatment had the highest effect on the 10% PC system showing up to 70% increase in flexural strength and the lowest effect was on the 18% polymer and 6% glass fibers (less than 10% increase). The effect of silane on the increase in flexural modulus of glass fiber reinforced PC is shown in Fig. 11.

Fracture Properties

The performance of PC materials are affected by cracks (15) and their growth during loading. Hence eight silane treated glass fiber reinforced PC beam specimens were tested in four-point bending to quantify the resistance of the material during crack growth. The notch-to-depth ratio varied from 0.26 to 0.80. The data points needed to plot the fracture resistance curves are shown in Fig. 12. Using the concept of linear elastic fracture mechanics (LEFM), the relationship between elastic crack mouth opening displacement (CMOD^e) and the corresponding crack length in four-point bending can be represented as (16)

$$CMOD^{c} = 4 \sigma a V(\alpha)/E'$$
(3)

where σ is the net stress (6M/bd²); M is the applied pure bending moment; a equal to $(a + H_0) / (d + H_0)$ with H_0 being the clip gage holder thickness; a is the crack length; E' is equal to E (modulus) for plane stress and E/(1 -n²)^{0.5} for plane strain where v is the Poisson's ratio. An empirical formula with 1% accuracy for any a is used to calculate V (a) and is expressed as

$$V(\alpha) = 0.8 - 1.7 \alpha + 2.4 \alpha^2 + 0.66/(1 - \alpha)^2$$
(4)

Hence if during slow growth CMOD^e could be determined at various loading levels by unloading the specimens, and hence using Equation (3) it would possible to determine the corresponding crack length. A numerical iterating procedure was used to determine the corresponding crack length a. Crack extension Da is equal to $(a - a_i)$, where a_i is the initial crack length. Assuming a beam of cross section bxd with effective crack length a, the stress intensity factor was calculated using the equation for the four-point bending developed by Brown and Srawley ¹⁷. The relationship is as follows:

$$K_{I} = 6 M a^{1/2} Y(a/d)/b d^{2}$$
 (5a)

where

$$Y(a/d) = 1.99 - 2.47 (a/d) + 12.97 (a/d)^2 - 23.17 (a/d)^3 + 24.80 (a/d)^4$$
(5b)

Glass fiber reinforced polyester PC shows substantial amount of nonlinearity up to peak stress and hence resistance curves (R-curves, ASTM E 561-81) are used to characterize the resistance to fracture during slow stable crack extension in such materials.

To construct the K_R curve, effective crack length based on the CMOD method was used. Once the load P and the corresponding effective crack length is known from the test records, the value of K_I was obtained from Equation (5). As shown in Fig. 12, K_R - Da relationship (solid lines) can be best expressed in the following linear form

$$K_{R} = K_{0} + \rho (\Delta a)$$
⁽⁶⁾

where K_0 , and ρ are parameters obtained from least square fit of the data. The parameter K_0 for 18% PC (treated blasting sand) with 2, 4, 6% fibers are 1.13, 1.16 and 1.15 MPa m^{0.5} and the values for r are 0.20, 0.56 and 0.43 MPa m^{0.5}/mm respectively.

CONCLUSIONS

The influence of glass fibers and silane coupling agent on a polyester based polymer concrete (PC) was investigated at room temperature. Based on the experimental study the following can be concluded:

- 1. Addition of glass fibers increased the flexural strength and the strain at peak stress but did not change the flexural modulus of the PC systems. The 18% PC with 6% glass fiber had a flexural strength of 33 MPa (4,800 psi), about 80% increase over the unreinforced PC system.
- 2. Use of silane treated aggregates and fibers further improved the strength of the PC systems. Silane treatment had the greatest effect on the 10% PC system. Silane treatment improved the flexural modulus of various PC systems. Silane treatment doubled the flexural strength (three-point bending) of 18% PC with 6% glass fiber to 41.6 MPa (6,040 psi).
- 3. The two-parameter relationship effectively predicted the flexural stress-strain relationships for the glass fiber reinforced and unreinforced PC systems.
- 4. Crack resistance curves based on stress intensity factor (K_R -Da relationship) have been found to be best approximated by linear relationships.

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Constituent material	Relative weight %
Matrix-Polyester Resin	10 - 18
Initiator-MEKP	1.5*
Promoter-Cobalt Napthenate	0.3*
Aggregate-Blasting Sand	82 -90

TABLE 1 — COMPOSITION OF POLYMER CONCRETE

MEKP-Methyl Ethyl Ketone Peroxide; *by weight of resin



Fig. 1-Particle size distribution of sand compared to ASTM C 33-85 for fine aggregates



Fig. 3-Variation of flexural strength of 14 wt percent PC on a normal probability plot