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MODELING AND EXPERIMENTAL CHARACTERIZATION OF STEEL FIBER/POLYMER INTERFACE: PULL–OUT TESTS

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ABSTRACT

In the current industrial scenario metal fibers reinforced polymers are used in many applications like car tyres and bumpers. These metal/steel fibers need perfect sizings compatible with the polymer giving very good interface properties for high end product performance. In the present work the interface properties between steel fibers and polyester resin are studied.

A single fiber pullout test has been developed for measuring the debond energy and frictional parameters for high carbon steel fibers embedded in a polyester matrix. The experimental setup implies a laser scanning extensometer for direct measurement of the pullout length. The pullout load measured consists of two components: a) debonding of the adhesive joint and b) frictional sliding of the debonded portion of the fiber. The hysteresis between unloading and reloading is characteristic of frictional sliding between matrix and fiber. Load-unload-reload loops are performed in order to determine the frictional behavior of the interface.

The experimental results are compared to the pullout model by Hutchinson and Jensen [1], which was later further developed by Marshall [2] to account for the unload-reload hysteresis. The experimental investigation of unloading and reloading behavior of fibers pulled out from the polymer is in good agreement with the model.

1. INTRODUCTION

As the performance requirements of industrial products are increasing beyond the capabilities of conventional materials for several engineering applications, designers increasingly switched towards the usage of composite materials. The properties of advanced composite materials depends on the constituent material selection, manufacturing method chosen, compatible fiber sizings, and interface design confirming the affordable composite structures far superior to metal products. Steel fibers are one among several fiber reinforcements available today in the market for composite product developments. Even though the density of steel fiber is much higher, compared to other fiber reinforcements (like glass, carbon, aramid, and natural fibers e.g. flax and hemp), it offers several advantages when combined with polymers in terms of processing and performance enhancements. Steel fibers are heavily used in building construction industry developing in to steel fiber reinforced cementitious composites for big and tall structures, and also in good use for car tyres and bumpers. Very few researchers tried to use the steel fibers in polymer composites field. In order to obtain the desired performance characteristics with polymers, an interface bond between steel fiber and polymer matrix plays a crucial role for better performance. To study the fiber-matrix adhesion strength, four standard micromechanical test methods are described in the literature. They are fragmentation, micro compression, micro
tension, and pullout (push out) tests. In the present work, fiber/matrix interface characteristics were evaluated by fiber pullout test.

Kharrat et. al [3] developed an analytical model which assumes an elastic load transfer between the fiber and matrix (shear-lag model) for the first phase of the pull-out test (before debonding). After debonding, the fiber is extracted from the matrix against friction. To analyze this phenomena an analytical model, which assumes Coulomb friction at the fiber-matrix interface and Poisson’s effects on both fiber and matrix, was developed for the extraction process of fiber from matrix. This model was verified by the experiments for both stainless steel/polyester and stainless steel/epoxy composite systems. According to this model, the interfacial shear strength and the compressive residual stress for stainless steel/epoxy composite system was much greater than that for the stainless steel/polyester composite system. Similarly few research articles describe the interfacial property evaluation methods of steel fiber embedded in a cementitious composites. Morrison et al. [4] proposed a fracture mechanics approach, which is applicable to any elastic fiber and matrix system, but the results of steel fibers in a cementitious matrix are only reported in the article. The fracture parameters are used to predict the behavior of steel fibers of varying dimensions during pullout tests. Sameer et al. [5] discussed in detail the fundamental understanding of bond and bond mechanisms of steel fiber/very-high-strength concrete composites with a standard interfacial bond test method. The bond-slip characteristics of four different steel fiber types embedded in very high strength concrete were investigated by considering significant parameters like fiber geometry, fiber embedment length, medium strength fibers, and varying embedment methods. The author proved that the mechanical effect of fiber geometry has the most influence on both peak load and total work. The increase in embedment length also increases the peak pullout load and total pullout work.

Levasseur et al. [6] explained the procedure for identification of interfacial parameters of SiC/Pyrex composite determined by pullout tests. Both unloading and reloading cycles and monotonic loading were performed and the response of load versus crack opening displacement of a monofilament bridging a matrix crack is measured and the experimental measurements are made by using a classical model developed by Marshall [2]. The literature study shows that interfacial properties of high carbon steel fiber embedded in a polyester resin for composite applications are still in the development stage with a lack of proper research studies. The basic physics and fundamental understanding of interfacial properties and a quantitative characterization of interfacial adhesion strength can help in evaluating the mechanical behavior and capabilities of macro scale composite structures. Hence, in the current research study a series of experiments were conducted and compared to the pullout model developed by Hutchinson and Jensen [1], which was later further developed by Marshall [2] to account for the unload-reload hysteresis.

2. MATERIALS AND MANUFACTURING

The test specimens for pullout test are difficult to manufacture. The placement of the fiber exactly at centre of polymer block was challenging. Another issue is polymer shrinkage and air entrapment which will cause several issues while processing the test specimen in open and closed moulds. In the current research study, identifying the best processing method and standard
procedure to make test specimens were considered. The materials and processing techniques used for developing the test specimen are discussed below.

### 2.1 Materials

The metal fiber/filaments used in the current research study are high carbon steel fibers with a fiber diameter 0.21mm. In order to embed the fiber in the thermoset polymer, a standard polyester resin is used. Polyester resin “Polylite 400-499” grade resin was considered for the current study. A silicone mould was designed having a cavity 5mm x 5 mm with a pin hole to place the fiber exactly at the centre of cavity.

### 2.2 Manufacturing Methods

Single steel fiber embedded in a polyester resin with rectangular cross-section are cast in silicone moulds, see Figure 1a. Cross sectional dimensions are 5 x 5 mm. The steel filaments are led through slots in the moulds, and they are kept straight by applying loads to the ends of the filaments. The process was optimized to provide specimens with straight fibers positioned in the centre of the specimens and without any air inclusions. The open mould specimens are not attractive due to lots of bubbles and polyester resin shrinkages. The specimen surfaces are getting concave shape due to resin shrinkage instead of obtaining flat surfaces on the final specimen.

![Figure 1. Single fiber pullout test specimens - casting in open and closed silicone moulds](image)

To improve the specimen quality without bubbles/voids observed inside the polyester, and also to eliminate the uneven surfaces such as concaveness, a closed mould process is chosen. A closed mould process is a vacuum infusion technique considered to improve the quality of the specimen. The process is simple and easy to maintain process parameters and help to produce the specimens with any other material combination with the same process conditions. The Figure 1b shown above is a double vacuum bag molding method with different vacuum level used in two
vacuum bags. The vacuum level used in inner bag is under full vacuum (100%), whereas the vacuum level in outer bag is around 40%. These two vacuum levels in the bags keep the setup air tight ending up with good infusion trials without any leaks or improper infusion. The setup ensures the steel fibers are placed exactly at centre of the mould and also fibers are in straight position. After the infusion of polyester resin into the mould and filling up the rectangular cavity, the setup should be kept in vertical position hanging the vacuum bag in a compatible stand. The resin is filled properly into the mould cavity avoiding resin rich regions at one end and resin less regions at other end of the mould. After finishing the infusion, the setup is kept overnight to complete the curing and solidify the polyester resin. The specimens were taken out from silicone moulds and checked for bubbles and the specimens were discarded if bubbles were present inside the specimen. The specimens were placed in oven around 60 degree C for few hours to remove the jelly/sticky surface. With this procedure the specimen were produced and used for further test trials.

3. EXPERIMENTS

The specimens made by vacuum infusion process were used to evaluate the interface characteristics between steel fiber and polyester resin. Two types of tests were considered to determine the interface parameters.

3.1 Fiber Pullout Test

Steel fiber pullout tests were performed similar to procedures described in literature [7]. In the current study, the setup shown in Figure 2a, was used for a simple pullout test. The filament/fiber is fixed on a capstan, whereas the polyester surface is supported at the top as seen in Figure 2b. Steel fiber is in tensile mode and the polyester resin block is in compressive mode during loading of the specimen. As the load applied on the specimen increases, the fiber gets elongated and the first phase of failure starts cracking the interfacial bond with the polyester resin from top surface to bottom end. During this phase the load/stress transfer takes place from fiber to polyester resin and the bond breaks slowly at the interface with the slight contraction of fiber and slight increase in the diameter of the hole where fiber was sticking to polyester surface. When the crack propagation ends, the second phase of failure starts. In this mode, the fiber is pulled out against the friction. The fiber movement can be seen clearly with movement from bottom end of the polyester surface. This continues until the fiber is completely pulled out from the polyester resin. During the pullout process, in the first phase the load starts from zero and reaches the maximum value (debond load) and from there the load drop takes place to initial extraction load, simultaneously with a jump of fiber movement initiating the second phase of failure called slip stick phenomena [8] (few cases observed a smooth pullout of fiber against the friction instead of slip stick) as shown in Figure 3.
Figure 2. Experimental setup – Pullout test

Figure 3. Typical pullout curve – load versus cycle elapsed time
3.2 Load/Unload/Reload Experiments

The load/unload/reload experiments were designed on single fiber pullout test specimens. The specimen is produced similarly to the test specimens used for simple pullout test described in the previous sections. The bottom end of the polyester material was fixed in a grip and top end of the steel fiber was fixed on the capstan. Since the polyester is in a fixed grip, the fiber as well polyester material were in transverse compression at the bottom end, as shown in Figure 4.

Test specimens used in the load/unload/reload experiments have 35 mm and 40 mm embedded steel fibers in polyester resin block of 5mm x 5mm with a fiber diameter 0.21mm. The test specimen was considered based on the embedded lengths of fiber. The present study deals with the identification of interfacial parameters from this modified pullout test. To determine the quantitative parameters, loading and unloading/reloading curves were considered as shown in Figure 5. When the loading initiates, the fiber gets elongated elastically at initial state due to tensile loads. During this period the interfacial crack initiates from top end. Before it reaches the other end of the specimen, unloading the test specimen creates reverse sliding. This sliding occurs along only a portion of the debonded region in the context of pullout. During reloading the test specimen the partial debond further extends along the fiber/matrix interface and travel to other end of the specimen as the peak load reaches the maximum debond load. After this process the second phase failure initiates causing frictional sliding. The stress/strain behavior is decaying to a plateau in the debonded region, giving a “reverse sliding” [8]. This sliding starts from the point where the fiber enters the resin and propagates along the debonded interface.

Figure 4. Test setup for load/unload/reload steel fiber and polyester specimen
4. ANALYTICAL MODELING

The modeling of fiber pullout phenomena during initiation of fiber-matrix debonding and sliding against the friction has been studied by several researchers. In the present work, to study the initiation of debonding and friction sliding basic models like shear lag model [9] and Coulomb friction models were considered [10]. Apart from this a pullout model developed by Hutchinson and Jensen [1], which was later further developed by Marshall [2] to account for the unload-reload hysteresis, were considered to determine the fiber-matrix interface parameters.

4.1 Initial Debonding

The fiber-matrix interfacial bond crack initiation, propagation, and termination during pullout process were analyzed by using the basic shear lag model [9]. The model assumes elastic load transfer between fiber and matrix. In the present work, to analyze the fiber/matrix interface, the derivations given by Kharrat et al. [3] were considered. The maximum fiber tensile stress $\sigma_{\text{Max}}$ can be expressed by maximum interfacial shear stress $\tau_{\text{iMax}} = \tau_{i}(0)$:

$$\sigma_{\text{Max}} = \frac{2.\tau_{\text{iMax}}}{\alpha.r} \cdot \tanh(\alpha.L)$$  \hfill (1)
When debonding initiates, $\sigma_{\text{Max}}$ attains $\sigma_d$ and $\tau_{i\text{Max}} = \tau_d$, which is defined as interfacial shear strength for debonding between fiber and matrix.

$$\sigma_d = \frac{2.\tau_d}{\alpha_r} \cdot \tanh(\alpha_r L) \quad (2)$$

$$\alpha^2 = \frac{4G_m \left[ \frac{r^2}{R^2} \left( \frac{E_f}{E_m} - 1 \right) + 1 \right]}{r^2 E_f \cdot \left[ 2.\ln \left( \frac{R}{r} \right) - \left( 1 - \frac{r^2}{R^2} \right) \right]} \quad (3)$$

Where $E_f$ is fiber modulus, $E_m$ is matrix modulus, $G_m$ is shear modulus of the matrix, $r$ is fiber radius, $R$ is the matrix cylindrical radius, $L$ is embedded length, $\sigma_d$ is debonding fiber stress, $\tau_d$ is interfacial shear stress.

### 4.2 Frictional Sliding

During fiber extraction process from matrix against friction, the traction state of stress allowed the fiber to contract decreasing its radius and matrix undergoes a compressive state of stress and its radial displacement increases at the interface [3, 11]. In this state of frictional sliding, Poisson’s ratio of fiber and matrix needs to be considered for evaluating interfacial frictional shear stress.

Fiber extraction stress: $\sigma_f(x) = \frac{\sigma_0}{K} \left[ 1 - \exp \left( -\frac{2\mu}{r} K(x + L) \right) \right] \quad (4)$

Initial tensile stress on the fiber: $\sigma_f = \frac{\sigma_0}{K} \left[ 1 - \exp \left( -\frac{2\mu}{r} KL \right) \right] \quad (5)$

$$K = \left[ \left( \frac{\nu_f}{1 + \nu_m} \right) \frac{E_m}{E_f} - \left( \frac{r^2}{R^2 - r^2} \right) \frac{\nu_m}{1 + \nu_m} \right] \quad (6)$$

Where $\mu$ is the coefficient of friction at the fiber-matrix interface, $\nu_m$ is Poisson’s ratio of matrix, $\nu_f$ is Poisson’s ratio of fiber, $\sigma_0$ is residual stress, $x$ is the bottom end fiber position i.e. fixed distance from top end to bottom end of fiber, and $L$ is embedded length.
Figure 6. Typical pullout curve – load versus displacement curve steel/polyester specimen (displacement rate 5mm/minute)

Figure 7. a) Debonding fiber stress versus embedded length for steel/polyester system
b) Debonding fiber stress versus fiber aspect ratio for steel/polyester system
Figure 8. Hysteresis loops – Single steel fiber reinforced polyester pullout specimen
4.3 Load/Unload/Reload Analysis

The analytical expressions of fiber pullout during load/unload/reload cycles were first derived and modeled in the literature by Hutchinson and Jensen [1], later studied by Marshall [2]. These models are derived based on the solutions of Lame’s theory. The theory was well suited for identification of interfacial parameters on a micro scale composite such as single fiber embedded in a polymer. Friction is modeled using Coulomb’s law considering the Poisson’s contraction effect and residual radial strains at the interface. The debonding mode of failure observed in pullout test was mode II interface fracture. Few important equations are given below for analyzing the test data, but the detailed derivations are shown by the authors [1-2] with proper explanation:

Normalized applied fiber stress: \( S_a = \frac{F}{\pi r^2 \sigma_p} \)  

For monotonic loading, \( \delta = \frac{\delta^1}{\mu} \left[ -AS_{R0} \ln \left( \frac{S_{R0} - S_a}{S_{R0} - \Gamma^1} \right) + \Gamma^1 - S_a \right] \)  

Effective radial clamping stress: \( S_{R0} = \frac{-\sigma_{r0}}{b_1 \sigma_p} \)  

For unloading: \( \delta_u - \delta = \frac{\delta^1}{\mu} \left( S_{R0} - S_a \right) \left[ 1 - \left( \frac{S_{R0} - S_a}{S_{R0} - S_{R0}} \right)^2 \right] \)  

For reloading: \( \delta - \delta_{re} = \frac{\delta^1}{\mu} \left( S_{R0} - S_a \right) \left[ 1 - \left( \frac{S_{R0} - S_{re}}{S_{R0} - S_{R0}} \right)^2 \right] \)  

Where \( \sigma_p \) is the maximum applied fiber stress, \( r \) is the fiber radius, \( F \) is applied load, \( \sigma_{r0} \) is a component of the radial stress at the sliding interface due exclusively to roughness and thermal misfit effects, \( b_1 \) is a constant parameter (refer [1-2]), \( \mu \) is the frictional coefficient, \( S_u \) and \( \delta_u \) are defined as the normalized stress and displacement at which an unloading sequence starts, similarly \( S_{re} \) and \( \delta_{re} \) are defined as the normalized stress and displacement at which an reloading sequence starts.

The above equations provide a relation between displacements \( \delta \) and applied load \( S_a \), with four other parameters which characterize the frictional properties of the interface: the fiber displacement (\( \delta^1 \)), the residual stresses (\( S_{R0} \)), the normalized debond energy (\( \Gamma^1 \)), and the elastic properties and misfit strain anisotropy of the fiber and the matrix (\( A \)).

5. RESULTS AND DISCUSSIONS

The above experimental trials and analytical modeling on single fiber pullout of steel filament reinforced in polyester material give preliminary knowledge on interfacial behavior of materials. In both the experimental trials such as simple pullout and unload/reload cycles, the displacement rate is assume as 5 mm/min. The experimental test results and analytical modeling results are summarized under following subsections.
5.1 Effect of Fiber Embedment Length on Shear Stress

As observed with special lenses, the debonding of the interface occurs in the initial linear portion of the curve. The debonding crack continues to propagate as the applied load is increased and completes the debonding at the point where the load reaches to maximum value called $F_d$. This is followed by a big drop in the load from $F_d$ to initial extraction load ($F_i$), this load level is required for pullout extraction of the debonded steel fiber from the polyester resin. The influence of fiber embedment length on interfacial shear stress of pullout test specimen is studied in the present work. The figure 6 represents the experimental data for steel fiber/polyester system with different embedded lengths i.e. 8mm and 12.5mm. The Table 1 shows the debonding load and initial extraction loads are higher for higher embedded lengths. For a composite system with a larger $F_d$ (debonding load) has higher shear strength. The interfacial shear strengths calculated by Kharrat model [3] using the experimental data for both specimens having different embedded lengths are given in Table 1. After completion of the initial interface debonding, both the specimen show unstable fiber pullouts with a slip-stick behavior. Figure 7a show the effect of embedded length over debonding fiber stress with a varying fiber diameter and resin dimensions. Similarly Figure 7b shows the effect of fiber aspect ratio on debonding fiber stress for steel fiber/polyester system with varying dimensions. The two curves in Figure 7b coincide because the fiber debonding stress, Equation (2), depends on the aspect ratio (rather than on the embedded length). Thus higher the interface strength, better the composite performance due to effective stress transfer from the matrix to the fibers.

5.2 Load/Unload/Reload Cycle Effects on Fiber Debonding and Sliding

A series of tests were conducted to investigate loading, unloading, and reloading the specimen to observe the pullout pattern and interface debonding between steel fiber and polyester. The hysteresis loops exhibit same shape but they are shifted on the load axis. The cross head speed displacement maintained constant 5mm/min for all the tests, which smoothes friction instabilities and the initial stress at the fiber interface. Due to small amplitude load fluctuations, the instabilities were indicating the rough sliding behavior as explained by Mumm and Faber [12]. Coulomb friction is regarded as the fundamental friction law operating at contacts. During unloading and reloading cycles the debonding cannot continue like a simple pullout behavior. The experimental data recorded for steel fiber/polyester specimen while loading, unloading and reloading are shown in the graphs (see Figure 8). The parameters that can be measured which relate to the steel fiber/polyester interface properties are shown in Figure 8a. In the first case, loading, unloading and reloading the specimen performed at 40N with a decrease in 10N for every cycle until it finally reaches 0N (the curve shown in Figure 8a correspond to first case). The hysteresis loops shown in all load drops are having same unloading modulus. Whereas in the second case, monotonic loading and several unloading/reloading cycles were performed at an increase of every 10N load and unloading to 2N continuing further to reloading next cycle until the fiber broke. Even in this case the slope of the unloading modulus for each cycle is same, but the hysteresis loop width increases as it goes to higher reloading cycle. The area of the hysteresis loop is the energy dissipated by the fiber at the interface and called as debond energy.

5.3 Determination of Interface Parameters
The laser extensometer used in the current test set up helps to find exact pullout lengths and fiber displacement. The parameters determined based on experimental data with the use of analytical model for a simple pullout steel fiber/polyester specimen are given in Table 1. It is interesting to see the interface strength values for steel fiber/polyester are comparable with glass/polyester composites. Similar to simple pullout, hysteresis that occurs during an unload/reload cycle relates to the sliding stress, can be evaluated from the experimental data. The parameters identified on unloading/reloading loops appear more reliable to characterize interfacial behavior only when sliding occurs during reverse sliding and debonding at the interface. The preliminary investigation results are given in the Table 2 and Table 3. The calculated value obtained by modeling are residual stress (2273MPa), normalized debond energy (-1.02), and dimension less parameter ‘A’ used in evaluating the properties, which derived from the elastic properties and misfit strain anisotropy of fiber and matrix (1.35). The residual stress is very high and debond energy is very low values obtained by the analytical model, the results are not reliable at this moment. Debond energy obtained by model is negative value, this indicates spontaneous debonding and sliding between the fiber and polyester during sectioning of the specimen [1-2].

Table 1. Interfacial properties of steel/polyester specimens by shear lag model

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Fiber diameter (mm)</th>
<th>Embedded length (mm)</th>
<th>Maximum debond load ( F_d ) (N)</th>
<th>Interfacial shear strength ( \tau ) (MPa)</th>
<th>Initial extraction load ( F_i ) (N)</th>
<th>Frictional shear strength (using eqn 5) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.21</td>
<td>8.0</td>
<td>17.4</td>
<td>19.1</td>
<td>8.7</td>
<td>1.65</td>
</tr>
<tr>
<td>2</td>
<td>0.21</td>
<td>12.4</td>
<td>24.6</td>
<td>19.1</td>
<td>15.5</td>
<td>1.89</td>
</tr>
</tbody>
</table>

Table 2. Data obtained by load/unload/reloading experiments

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Fiber diameter (mm)</th>
<th>Embedded length (mm)</th>
<th>Unloading modulus [GPa]</th>
<th>Permanent strain (%)</th>
<th>Experimental data</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.21</td>
<td>35</td>
<td>100</td>
<td>0.2</td>
<td>Figure 8b</td>
</tr>
<tr>
<td>2</td>
<td>0.21</td>
<td>40</td>
<td>113</td>
<td>0.4</td>
<td>Figure 8a</td>
</tr>
</tbody>
</table>

Table 3. Interfacial parameters obtained by modeling the load/unload/reloading tests

<table>
<thead>
<tr>
<th>Sl. No.</th>
<th>Normalized residual stress ( S_{R0} )</th>
<th>Coefficient of friction ( \mu )</th>
<th>Residual stress ( \sigma_{R0} ) [MPa]</th>
<th>Normalised interfacial debond energy ( \Gamma^{-1} )</th>
<th>Dimension less parameter ( A )</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>2.95</td>
<td>0.35</td>
<td>-2273</td>
<td>-1.02</td>
<td>1.35</td>
</tr>
</tbody>
</table>
6. CONCLUSIONS

The pull-out behavior of steel fiber and polyester resin is investigated by both experimental and analytical approaches. The experimental investigation of unloading and reloading behavior of fibers pulled out from the polymer is studied with respect to the analytical model. Based on the experimental and analytical results introduced in this paper, several conclusions can be drawn and summarized in the following.

(1) Test specimen produced under closed mould method (vacuum infusion technique) is much better than the open mould method.
(2) Under equivalent conditions, the failure mode and pull-out strength of steel filament are significantly influenced by the embedded length of fiber.
(3) Two phases of failure mode were observed in the tests. Initiation of interfacial crack and termination of crack while reaching maximum pullout loads, whereas the second phase of failure is pullout of the fiber against frictional sliding.
(4) Both shear lag and Coulomb friction models developed by Kharrat [3], used to predict the interfacial properties, which are in good agreement with the experimental values.
(5) For specimen with long embedded fiber under repeated loading, the ultimate strength keeps the same level with the ones under monotonic loading.
(6) Laser extensometer used in load/unload/reload experiments to record the exact fiber displacements and debond lengths improves the experimental data.
(7) The load-displacement relationships for unload/reload cycles obtained from the analytical model are in good agreement with the experimental data as well the debonding crack propagation patterns. The analytical results show that the experiments can help in evaluating the interface characteristics.

The present article is a preliminary research study for steel fiber and polymer interface characterization. The work is in progress and the future work focuses on simulation of test results with the numerical modeling. This can confirm the results obtained in the preliminary investigation are correct and can be used for analyzing material system at macro level composites.

7. ACKNOWLEDGMENTS

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8. REFERENCES


