

Creep of Fiber Reinforced Polymers-Epoxy-Concrete Interface Incorporating Carbon Nanotubes

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Fiber reinforced polymer (FRP) composites are widely used in structural strengthening and retrofitting for existing reinforced or prestressed concrete structures due to their high strength-to-weight ratio and non-corrosiveness. However, one of the drawbacks of the FRP composites is the relatively high creep deformation of epoxy when used to bond FRP sheets to concrete structure against sustained loads. On the other hand, carbon nanotubes (CNTs) are reported to provide significant enhancement to various mechanical properties when used with epoxy. This enhancement is attributed to the extra-ordinary mechanical properties of the CNTs.

In this article, we report the results for a recent experimental investigation conducted to examine the long-term behavior of CNTs-epoxy composites used at the FRP-concrete interface. Double shear tests were performed on FRP sheets bonded to concrete blocks using CNTs-epoxy composites. Various levels of CNTs by weight were examined including 0.5%, 1.0%, and 1.5%. The CNTs were dispersed in the epoxy matrix, tested, and creep was measured and compared with the case of epoxy without CNTs. The results show the ability of CNTs to produce a limited reduction in creep compliance of epoxy at the FRP-concrete interface.

1 INTRODUCTION

Retrofitting and strengthening of existing reinforced or prestressed concrete structures have been a rapidly growing area in civil engineering in the last three decades. One of the excellent techniques developed over years are strengthening using fiber reinforced polymer (FRP) composites due to their high strength-to-weight ratio and durability. Researchers and designers reported many successful field applications where FRP was used successfully in new structures or in strengthening of existing structures. However, on some occasions, this technique might experience premature failure if lack of shear transfer at the FRP-concrete interface takes place and that might lead to debonding. Furthermore, viscoelastic behavior (e.g. creep) of adhesives at the interface was reported recently to be problematic when FRP strengthening is used to support sustained loads (Reda Taha et al. 2010). Recent experiments by Reda Taha et al. (2010) on full scale beams showed that creep of the epoxy adhesive can result in off-loading the FRP strips and thus late cracking of concrete. The double lap shear test, proposed by the Japanese Concrete Institute, is examined by Ferrier and Hamelin, (2002) to examine the creep behavior of FRPconcrete interface as a function of time and temperature and estimate the corresponding rheological model parameters. Feng et al. (2005) studied the creep behavior of structural adhesives using accelerated creep tests. They concluded that the long-term creep behavior of the epoxy adhesives can be reliably predicted using a set of short term accelerated creep tests. Benyoucef et al. (2007) developed a closed form solution to describe shear creep and shrinkage of adhesives at the FRP-concrete interface. Based on a theoretical approach, it was found that



edge interfacial stresses decrease as the thickness of the adhesive increases. It was also observed that creep of adhesives increases the bond length of the FRP significantly (Wu and Diab 2007). Meshgina et al. (2009) performed extensive study to examine the effect of different parameters on creep of epoxy at the FRP-concrete interface using double shear tests. It was found that the epoxy thickness, curing time, and load level significantly affect epoxy creep. In addition, Majda and Skrodzewich (2009) examined the creep of epoxy adhesive under tension and showed using rheological models that the stress to strength ratio has the most significant impact on creep behavior. Furthermore, Hamed and Bradford (2010) found that creep causes significant shear and normal stresses of the adhesives at the edges. Recently, Ferrier et al. (2010) examined time-temperature principle on epoxy adhesives with various glass-transition temperatures (T_g) subjected to shear loading. It was concluded creep is correlated to T_g temperature of epoxy.

Since their discovery by Ijima (1991), carbon nanotubes (CNTs) have been a target for research by many scientists worldwide to investigate their properties and develop techniques to increase their use. Currently, CNTs mechanical and electrical applications include polymer, metal and ceramic composites, scanning probe tips, and contact devices for micro electro mechanical systems (MEMS) (Eklund et al. 2007). CNTs are known to have properties of one or more order of magnitudes higher than any existing structural materials. For example, CNTs Young's modulus is 10-100 times higher than the strongest steel (Advani and Fan 2007). Large portions of the CNTs research are oriented to their use with polymers in nano-composites. CNTs were found in many investigations to enhance various mechanical properties of polymers such as stiffness, strength and toughness (Spitalsky et al. 2009). However, the time dependant behavior of CNTs-polymer composites is rarely examined. Suhr et al. (2005) examined the viscoelastic behavior of thin epoxy film with Multi-Walled Carbon Nanotubes (MWCNTs) under shear and observed significant increase in damping ratio. Also, Zhang et al. (2007) examined creep behavior of CNTs/epoxy composite under tension and observed reduction in creep strain up to 30% with 0.1% Single-Walled Carbon Nanotubes (SWCNTs). Significant enhancement in creep resistance of CNTs/epoxy composite using magnetic field was found by Tehrani and Al-Haik (2009). In this study, double lap shear test was performed to investigate the effect of incorporating CNTs in epoxy on shear behavior of the FRP-epoxy-concrete interface.

2 METHODS

The double lap shear test was used to evaluate creep of epoxy with CNTs at the concrete-epoxy interface. The double lap shear test specimen consisted of two carbon fiber reinforced polymer (CFRP) composite laminates bonded to two sides of two concrete blocks using controlled thickness epoxy adhesive as shown in Figure 1. The concrete blocks were loaded in tension to induce shear stresses at the four epoxy joints. The applied load (*P*) induces shear stress of 25% of the ultimate shear strength of the epoxy. DEMEC (gauge) points were bonded to the concrete blocks and were used to measure the deformation of two shear joints in the loading direction. Unidirectional CFRP laminates were chosen in order to ensure minimal creep response of the FRP when loaded in the fiber direction. In addition, concrete blocks were cured for 7 days and left to dry for 21 days before being used in the experiments. The axial stress in concrete during experiments did not exceed 0.36 MPa (less than 10% of the tensile strength of concrete). Therefore, creep and shrinkage of concrete during the experiment was expected to be negligible compared with epoxy creep. By neglecting creep and shrinkage of concrete and creep of the CFRP laminate and ignoring the deformation through epoxy thickness, epoxy shear strain $\gamma(t)$ per joint can be computed as:

$$\gamma(t) = \frac{\Delta L(t)}{h} \tag{1}$$



Where (*h*) is the epoxy thickness and (*t*) is the time and $\Delta L(t)$ shear displacement per joint. In addition, the shear stress (τ) acting on every shear joint can be computed as follows:

$$\tau = \frac{P}{2A} \tag{2}$$

where (A) is the area of one shear joint. The normalized creep compliance $J_n(t)$ of epoxy is also computed relative to the elastic deformation using:

$$J_n(t) = \frac{1}{J(0)} \frac{\gamma(t)}{\tau}$$
(3)

where J(0) is the elastic compliance and $\left(\frac{\gamma(t)}{\tau}\right)$ is the creep compliance.



Figure 1. Schematic of the double lap shear specimen (a) dimensions and (b) deformation.

3 EXPERIMENTAL DETAILS

3.1. Material Properties

Table 1 shows the mix proportion by weight of the concrete substrates used in all experiments. The chosen concrete mix has a 28 day compressive strength of 42 MPa similar to most concrete used nowadays in construction of reinforced or prestressed concrete elements. MWCNTs were supplied by Cheap Tubes, Inc. and were used as-received. Based on the manufacturer's specifications, the CNTs were multi-walled with outer diameter (OD) of 20-30 nm, inner diameter (ID) of 5-10 nm and length of 10-30 μ m with an aspect ratio of 500 to 1000. The epoxy used in fabrication is EPOTUF[®] 37-127 epoxy system supplied by U.S. Composites, Inc. The epoxy resin is low viscosity, 100% reactive diluted liquid based on Bisphenol-A containing glycidyl ether. The hardener is Aliphatic Amine EPOTUF[®] 37-614. The resin to hardener mixing ratio is 2:1. The low viscosity of the resin facilitates incorporating the CNTs during the fabrication process and provides reasonable level of deformations in order to obtain accurate creep measurments. CFRPs composites were supplied by Graphtek LLC. The CFRPs were 1.1



mm thick uni-directional laminates fabricated using 33 m.s.i carbon and Vinlester resin.

Material	Content
Water (kg/m ³)	150
Cement (kg/m ³)	360
Class F Fly Ash (kg/m ³)	90
Fine Aggregate (kg/m ³)	920
Coarse Aggregate (kg/m ³)	809
Superplasticizer (mL/m ³)	7650
7 day compressive strength (MPa)	34.6 ± 2.2
28 day compressive strength (MPa)	42.6 ± 3.0

Table 1. Concrete mix proportions for concrete blocks used as substrate for FRP

3.2. CNTs dispersion

One of the major challenges of incorporating CNTs in polymers is to obtain a uniform dispersion of the CNTs. The CNTs dispersion difficulty arises due to van der Waals forces which might entangle the individual nanotubes to each other. Over the last decade, several techniques were developed to obtain a homogeneously seperated CNTs in the polymer suspension and to avoid the formation of CNTs agglomerations. In this study, physical approaches were implemented for their ability to provide simple and practical methods that can be implemented in the field with current FRP strengthening technology. A combination of ultrasonication and magnetic stirring were utilized herein (Figure 2(a-b)). The dispersion was performed by adding the required CNTs content to the epoxy resin and the CNTs-resin mixture was sonicated in ultrasonic bath for 1 hour at room temperature. Magnetic stirring was then applied to the dispersion for 30 minutes followed by 15 minutes sonication. The hardener was applied afterwards to the CNTs-resin dispersion and used to fabricate the creep specimens. The CNTs dispersion can be evaluated using scanning electron microscope (SEM) images.

3.3. Fabrication and Testing

In order to examine the creep of FRP-concrete interface, four different double shear specimens were fabricated and tested under sustained loads. The first specimen was fabricated with neat epoxy without CNTs while the other three were fabricated with CNT-epoxy composites with various CNTs percentiles. Three contents of CNTs by weight of epoxy were examined: 0.5, 1.0, and 1.5%. In addition to the creep specimens, similar specimens with neat epoxy were fabricated and tested to failure to obtain the ultimate shear strength of the joint. $50.8 \times 50.8 \times$ 127 mm concrete blocks were cast and cured in a water bath for one week. After wet curing, the concrete was left to dry for three weeks. $50.8 \times 50.8 \times 12.5$ mm steel blocks were bonded to the ends of the concrete blocks. This steel-concrete bond was left to cure for one week. The prepared CNTs-epoxy composites were cast on 25.4×25.4 mm shear joint. Glass beads with 0.71 ± 0.035 mm diameter known as class VI soda-lime glass beads supplied by Mo-Sci Corporation were used to control the epoxy thickness as recommended by ASTMs standards (ASTM D5868-01 2008). 0.5% glass beads by volume fraction of the epoxy were added to the concrete surface and the CNTs epoxy composites were applied afterwards by syringe pump to minimize the air voids at the shear joint. A pressure of 34 kPa was applied after placing the CFRPs composite over the epoxy shear joint and the excess of epoxy was carefully removed.



The shear joint was cured for 10 days before applying the sustained load. DEMEC strain gauges supplied by Mayes Instruments, Ltd were installed using a reference bar and dial guage was used to obtain strain mesurments during the creep experiment.





(a) Sonication of CNTs in epoxy resinFigure 2. Preparation of CNTs-epoxy composite.

(b) Magnetic stirring of CNTs-resin dispersion

The specimens were then loaded in a creep loading frame. Figure 3 shows the loading frame of the creep experiments. The frame consists of applying the sustained loads through steel weights. The applied load was measured using load cell (Figure 4(a)) and the load level of 934 ± 22 N was maintained at the specimen line of action. This load level induces shear stress of 724 ± 17 kPa at each shear joint which corresponds to 25% of the ultimate shear strength. A system of steel hooks and chains were used in transferring the load to the double shear specimens to avoid inducing any eccentricities. The creep experiments were performed under controlled environment of relative humidity (RH) of 60% and temperature of 23 °C. Figure 4(b) shows the four creep specimens attached to the loading frames. Finally, the ultimate shear strength test was performed using MTS[®] Bionix servo hydraulic system. The double shear specimens were loaded to failure by displacement control mode with loading rate of 0.5 mm/minute. Morover, single lap shear test of CFRP-CFRP interface bonded with neat epoxy was tested to failure.



Figure 3. Schematic of creep test setup.

4 RESULTS AND DISCUSSIONS

Figure 5(a-b) shows the failure of ultimate shear strength test specimens. It can be noted that the double lap shear test exhibited shear failure in the concrete substrate. Therefore, the shear strength of epoxy will not govern the concrete-FRP joint. On the other hand, the single lap shear test at which the failure occurred at the FRP-FRP interface and is considered more accurate in estimating the ultimate shear strength. The estimated ultimate shear strength from the double



and single lap shear joints are 2822 and 2521 kPa, respectively. A choice was made afterwards for the creep shear stress to be 25 % of the ultimate shear strength.





(a) Load cell attached to determine creep load(b) Four creep specimens loadedFigure 4. Creep experiment performed on four specimens.



(a) Double shear CFRP-concrete interface Figure 5. Failure of lap shear joints.



(b) Single shear CFRP-CFRP interface

Elastic shear strains per one lap shear joint for neat 0.5, 1.0, and 1.5% CNTs percentages were 1700, 1100, 2000, and 2100 microstrains respectively. This observation demonstrates that the highest elastic shear stiffness observed was in the case of 0.5% CNTs and the lowest was that with the 1.5% CNTs. In addition, the normalized creep compliance was computed and is shown in Figure 6. In general the case of 0.5% CNTs exhibited the lowest normalized creep compliance. As the CNTs content increased to 1.0 and 1.5%, the normalized creep compliance increased. Normalized creep compliances of 4.28, 3.75, 4.7, and 4.8 were observed after 10 days of measurements for the neat epoxy and epoxy incorporating 0.5%, 1.0, and 1.5% CNTs respectively. The maximum reduction of normalized creep compliance was observed at the 0.5% CNTs and was about 12% of the creep compliance observed in neat epoxy. Creep compliance reduction was therefore limited. The enhancement of the creep behavior associated with the 0.5% CNTs can be attributed to the CNTs resistance to long-term deformation of the polymer chains under sustained loads. As the CNTs content increases, difficulties in dispersion arise due to the formation of CNTs agglomerations. The agglomerations are expected to limit the elastic and long-term resistance of the CNTs-epoxy composite. The normalized creep compliance seems not to change beyond 1% CNTs. The mechanical enhancement using CNTs seems to be counteracted by agglomeration problems associated with using high CNTs content.

To further explain our observations, SEM investigations of the different CNTs-epoxy mixtures were performed. SEM images are shown in Figure 7(a-b). Two SEM images are presented in Figure 7 with the same length scale. In the case of 0.5%, randomly dispersed CNTs are



observed as bright spots within the epoxy matrix (Figure 7(a)). The size of these features is in agreement with the size of the nanotubes reported by the manufacturer. On the other hands, the formation of CNTs agglomerates in the case of 1.0% is evident as shown in Figure 7(b).



Figure 6. Normalized creep compliance of FRP-concrete interface for a period 10 days.





(a) Dispersed CNTs for the case of 0.5% Figure 7. SEM images for CNTs-epoxy composite.

(b) CNTs agglomerations in the case of 1.0%

In addition to the dispersion difficulties, the limited enhancements are attributed to the weak interfacial bond between the CNTs and the surrounding matrix. Perhaps providing chemical functionalization would improve the interfacial bond of the CNTs, which would lead to low creep compliance. Further research is warranted to examine the significance of functionalization on creep of epoxy. Examination of other CNTs content (especially lower than 0.5%) on the long-term behavior of FRP-epoxy-concrete interface seems necessary.

5 CONCLUSIONS

Creep experiments to examine creep of the FRP-epoxy-concrete interface were performed on double lap shear test specimens. Ultimate shear strength was determined using two different techniques. The FRP-to-FRP single lap shear test seems to be more accurate in estimating the ultimate shear strength than the FRP-concrete double lap shear test for the later being governed by concrete rather than FRP failure. The effect of incorporating various contents of CNTs is investigated. The case of 0.5% CNTs exhibited higher enhancement in elastic and creep deformations than the neat case. Degradation in creep behavior was observed in higher percentages of CNTs due to the formation of agglomeration. SEM images confirmed the



formation of agglomerates in 5.0 μ m length scale for the case of 1.5%. On the contrary, a uniform dispersion was observed in the case of 0.5%. Further investigation for the use of functionalized CNTs and lower CNTs content (less than 0.5%) seems necessary.

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