Fracture toughness and failure mechanism of high performance concrete incorporating carbon nanotubes

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ABSTRACT. Cement and concrete composites are inherently brittle and exhibit very less tensile/flexural strength capacity as compared to their compressive strength. Use of thoroughly dispersed carbon nanotubes in the concrete matrix is one of the possible solution for enhancing mechanical properties in tension/flexure. In the present research work, small fractions of multiwall carbon nanotube (MWCNTs) i.e. 0.05 and 0.10 wt% of cement have been integrated into the cement concrete to study their effect on the mechanical properties of the resultant concrete mixtures. The enhanced performance of the whole mix lies on a single point that MWCNTs must be thoroughly disperse in the mixture. Hence, special arrangement through usage of high energy sonication along with amended acrylic based polymer (performing as a surfactant) was made to have a uniform dispersion of MWCNTs in the concrete mix. The testing of concrete samples includes i.e., flexure, splitting tensile and compressive strengths after 3, 7, 28 and 56 days of curing. After having comparison with the control mix cured for 28 days, it was observed that the addition of 0.05 wt% MWCNTs increased the splitting

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tensile strength by 20.58%, flexural strength by 26.29% and compressive strength by 15.60%. Through above results, which verify the increase in concrete mix strength after adding MWCNTs, these MWCNTs may be incorporated in the treatment of Nano/micro cracks completed through process of connecting, branching and pinning. Similarly, as proved in three-point bending tests, MWCNTs also enhances the breaking strains as well as the fracture energy of the concrete mixes, besides, imparting increase to the strength. The investigations have shown that incorporating lesser amounts of MWCNTs i.e., 0.05 and 0.10 wt% of cement to the concrete mixes after insuring there complete dispersion, unusually improve their properties like mechanical strengths and fracture behavior.

KEYWORDS. Concrete; Fracture energy; MWCNTs; Toughness; Critical pullout length; Micro cracking.

INTRODUCTION

Cement and concrete composites are the basic construction materials which are extensively used around the globe [1]. The production of cement involves generation of enormous amounts of anthropogenic carbon dioxide (CO_2) in the atmosphere, contributing approximately 5.0% CO_2 generation around the globe. Beside this, other environmental concerns are also associated with the use of cement and concrete composites such as depletion of virgin aggregates and its impact on the ecosystem [2]. Ordinary cement and concrete composites offer much flexibility and cost effectiveness in their utilization but they are vulnerable to physical and chemical attacks affecting their performance in service life span; therefore, requiring costly repair and maintenance works. Construction of super-paves, tunneling, long span structural members and pre-stress technology demand the concretes of ultra-high strength and performance. For effective service life in different situations and under different loading conditions, ordinary concrete is not much beneficial. Therefore, the production of modified concrete with exceptional properties in terms of mechanical strength and with minimum amount of cement is highly desired so that economical and sustainable construction may be achieved along with reduction in CO_2 emissions in the atmosphere [3]. The idea of nanotechnology for the modification of composite properties at nano scale is not new in relation to the construction materials. Nanotechnology deals with the synthesis, characterization, utilization and analysis of materials at nano scale [4]. Several researchers have explained that the properties of cement and concrete composites may greatly be modified by using nano and micro sized particle inclusions in the matrix [5–12]. The nano level inclusions in concrete have shown improved durability, mechanical strength, porosity reduction and economical construction [13–19]. The nano metric inclusions includes nano silica, graphene, multi walled carbon nano tubes (MWCNTs), nano $CaCO_3$, nano TiO_2 etc. [20–26]. The studies show that these inclusions not only improve the packing of particles but also produce crack bridging phenomena by densifying the nanostructures [18,27]. Nano particles control the C-S-H reaction and improves the concrete durability [28–33].

Among above mentioned nano materials, MWCNTs possess unique and exceptional characteristics in terms of physical and mechanical properties. MWCNTs have tubular structure composed of folded layers of graphene with exceptionally high aspect ratios [34–37]. Several researchers have reported the utilization of MWCNTs in preparing cement and mortar composites and studied the behavior but limited work is available describing the full-scale utilization of the MWCNTs in the concrete matrix. Therefore, in the present research, MWCNTs were utilized in the preparation of concrete matrix and their influence on the mechanical behavior of concrete is discussed in detail.

EXPERIMENTAL PROGRAM

Materials

The concrete mixes were prepared from ordinary Portland cement (ASTM Type 1 grade 52.5), having specific gravity of 3.10. Locally available sand having fineness modulus of 2.13 and water absorption of 2.87% was utilized. Crushed lime stone aggregates confirming to the ASTM C33 were incorporated in the concrete mix. The characteristics of MWCNTs used for the preparation of concrete samples are presented in Tab. 1.

Diameter (*nm*)	Length (*μm*)	Purity (%)	Surface area (m^2/g)
6-25	10-50	>90%	250-300

Table 1: Physical characteristics of MWCNTs.

Dispersion of MWCNTs

The dispersion of MWCNTs was achieved with the help of bath sonication in the presence of modified acrylic based surfactant. For attaining good dispersion of MWCNTs in water, a solution of surfactant and water was prepared and then the measured amount of MWCNTs was added to the solution. The solution was sonicated for 20 min at 25±5°C. The dispersion was assessed qualitatively by filling test tubes with dispersed MWCNTs solution and observing the color of solution for next 48 h. The observations revealed that the MWCNTs did not settle down in the test tube and remained in the solution indicating effective dispersion of MWCNTs. The composition details of solution containing dispersed MWCNTs are presented in Tab. 2.

Designation	Water (*g*)	Surfactant (*g*)	MWCNTs (*g*)	Comments
R	100	2.0	0.000	Reference mix Mix containing 0.00% MWCNTs by mass of cement
CNT0.05	100	2.0	0.125	Mix containing 0.05% MWCNTs by mass of cement
CNT0.10	100	2.0	0.250	Mix containing 0.10% MWCNTs by mass of cement

Table 2: Composition of solution containing dispersed MWCNTs.

Mixture proportioning and sample preparation

The sand, aggregate and cement were mixed in dry condition as per the required quantities mentioned in Tab. 3. After dry mixing half of the water containing MWCNTs was added into the mixing machine and mixing was continued for 120 seconds at slow speed. After slow mixing, the machine was stopped for 30 seconds and walls of the machine were cleaned by trowel to separate any material attached to them. Then remaining water was added into the mix and the mixing was continued for 120 more seconds at high speed to achieve workable concrete mixture [38]. The mix proportions of concrete containing MWCNTs are reported in Tab. 3.

Designation	Cement (kg/m^3)	Sand (kg/m^3)	Aggregate (kg/m^3)	Water (kg/m^3)	Surfactant (kg/m^3)	MWCNTs (g/m^3)
R	476	690	1047	190.40	3.81	-
CNT0.05	476	690	1047	190.40	3.81	238
CNT0.10	476	690	1047	190.40	3.81	476

Table 3: Mix proportions of concretes containing MWCNTs.

After complete mixing, the concrete was poured into standard cylinders having 150 mm diameter and 300 mm height and beam molds of 100 mm x 100 mm x 400 mm. The molds were then kept in closed containers having 90% humidity for 24 h. The dried samples were then removed from the molds, labeled, weighed and cured in water at 25±2°C until the day of testing. The beam samples were notched with water cooled diamond saw blade and tested in three-point bending under crack mouth opening displacement (CMOD) controlled mode. The rate of CMOD was kept at 0.50 mm/min. The schematic diagram of test setup and sample geometry are shown in Fig. 1.

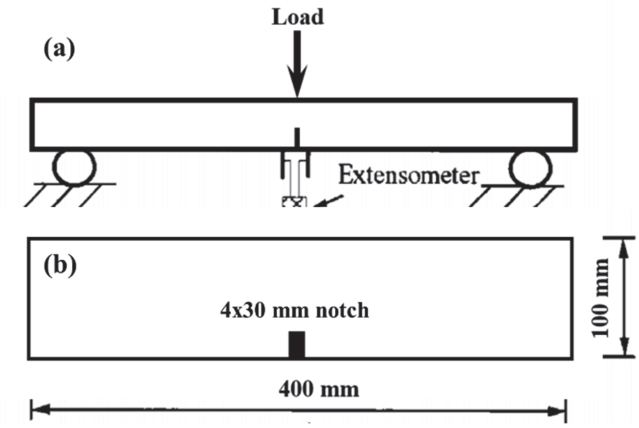

Figure 1: Schematic diagram of (a) Test setup; (b) Specimen geometry.

RESULTS AND DISCUSSION

Performance evaluation of samples in tension and flexure

The cylindrical specimens were tested as per ASTM C496 for determining their splitting cylinder tensile strengths at 3, 7, 28 and 56 days of curing under constant rate of loading i.e. 0.8 MPa/min. The test results are presented in Fig. 2 (a) below. The results revealed that the mixes containing MWCNTs exhibit higher tensile strength as compared to the plain concrete samples. Maximum enhancement around 26% was observed for CNT0.05 at 56 days of age whereas, it was around 18% for CNT0.10. The overall trend shows that the addition of small amounts of MWCNTs produce better results. This behavior may be attributed to the effective dispersion of MWCNTs at lower percentage addition. Whereas, in case of enhancement of compressive strength of modified samples the direct relation between the amount of MWCNTs inclusion and modification of strength was observed. It reveals that the reduction of effective water content ultimately results into the improvement in compressive strength. Dispersion concerns and agglomeration sometimes cause a reduction in the strength with increase in the quantity of MWCNTs [39].

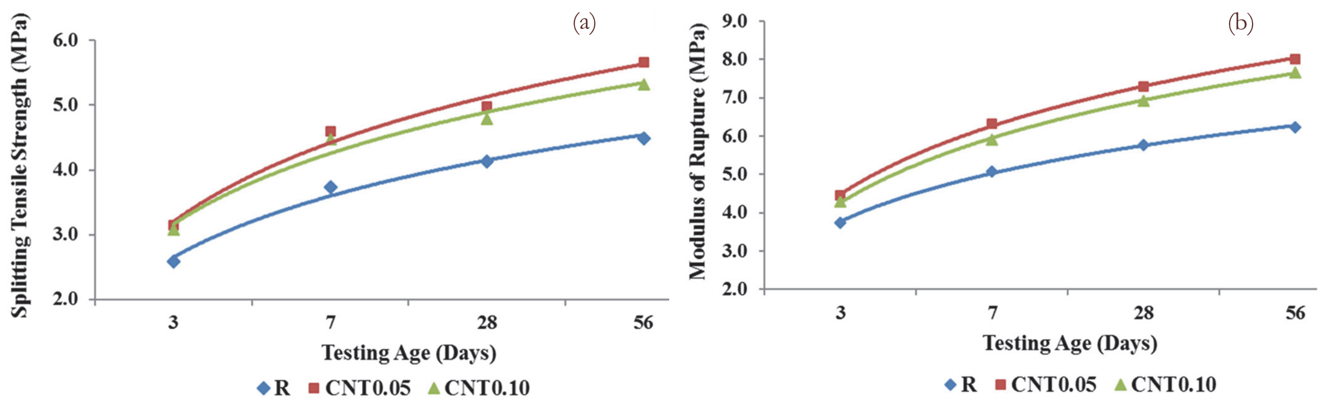

Figure 2: (a) Splitting cylinder tensile strength of concrete mixes; (b) Modulus of rupture of concrete mixes.

The three-point bending tests showed similar trend as produced by the splitting cylinder tensile tests. Overall better performance was achieved at 0.05wt.% addition of MWCNTs. A typical stress vs. crack mouth opening displacement (CMOD) curve is presented in Fig. 3, where it can be seen that the MWCNTs not only enhances the modulus of rupture but also substantially prolongs the post peak response of the specimens under investigation. The extended post peak behavior of concrete reinforced with MWCNTs is due to the crack bridging phenomena of MWCNTs, which imparts ductility and toughness.

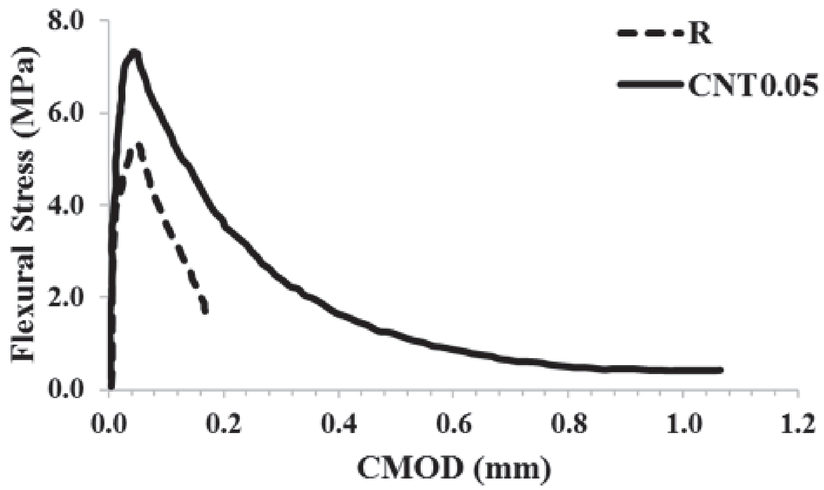

Figure 3: Typical stress vs. CMOD curves of concrete specimen with and without MWCNTs.

Performance evaluation of samples in compression

The concrete samples were also investigated for their compressive strength at 3, 7, 28 and 56 days of curing to assess the influence of MWCNTs addition on compressive behavior of concrete. The results of the compressive strength tests are presented in Fig. 4. The results indicate that the mix containing higher amount of MWCNTs exhibit higher compressive strength as compared to others. At 56 days of curing, the mix CNT0.10 gives 24.66% higher compressive strength than the control mix whereas; the enhancement was 19.11% for the mix CNT0.05. Similar pattern can be observed for strength at other ages.

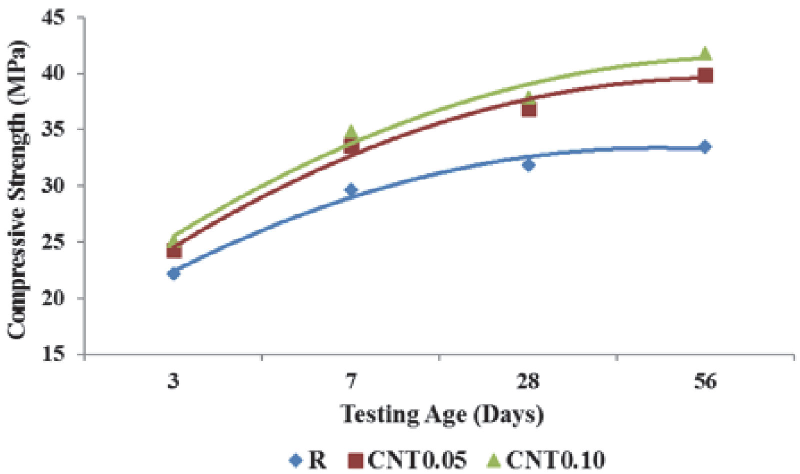

Figure 4: Compressive strength of concrete mixes with and without MWCNTs

Unlike flexural and tensile behavior, where better performance was observed at 0.05 wt% addition of MWCNTs; here in compression, the performance improves with the increase in the MWCNTs addition. This behavior may be attributed to the reduction in water content (i.e. effective w/c ratio) in the concrete mix due to the presence of large number of MWCNTs.

Strength activity indices of concrete mixes

For relative comparison of the influence of MWCNTs addition on the concrete mixes strength activity indices were evaluated and reported in Fig. 5 (a, b & c) below. The observations reveal that the concrete mixes containing 0.05 wt% MWCNTs perform better in splitting tensile strength and modulus of rupture whereas, concrete containing 0.10 wt% addition of MWCNTs perform better in compression as explained earlier.

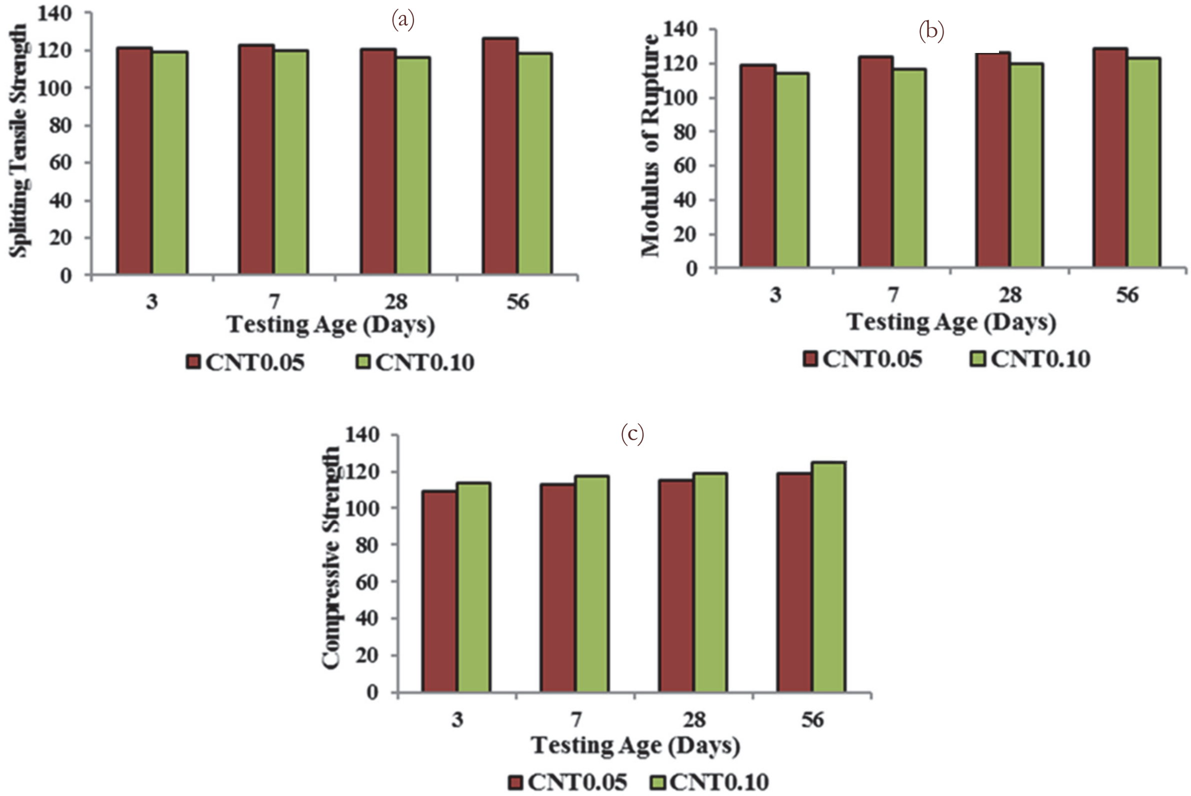

Figure 5: Strength activity indices for concrete mixes containing MWCNTs (a) Splitting tensile strength, (b) Flexural strength, and (c) Compressive strength.

Theoretical analysis of composite behavior

The mechanical performance of MWCNTs reinforced cementitious composites strictly depends upon their volume fraction, level of dispersion and average center-to-center distance in the matrix. According to the fiber spacing theory of Romualdi, the center to center distance between the inclusions is inversely related to the resistance offered by the composites to the progression of cracks [40]. This theory has been validated by several researchers and this may explain the phenomena of lower performance of cementitious composites in flexure containing higher amounts of MWCNTs [41,42]. When a flexural load is applied on the MWCNTs reinforced specimen, the load is transferred to the individual MWCNTs making them pull out from the matrix providing improved flexural strength and fracture energy. The improved tensile strength of the reinforced matrix may be computed by using following expression (Eq.1) which is further related to flexural strength of concrete specimen as presented in Eq.2 [43].

$$\sigma_{fc}^{t} = 2\eta_l \eta_o \left(L_c / d_c \right) \tau V_c \tag{1}$$

$$\sigma_{fc}^{t} = 0.41 \sigma_{fc}^{b} \tag{2}$$

where, σ_{fc}^{t} is the tensile strength of the reinforced composite, η_l is the coefficient of MWCNTs length, η_o is the orientation coefficient of MWCNTs, L_c is average MWCNTs length, d_c is the average MWCNTs diameter, τ is average bonding strength and V_c is the volume fraction of MWCNTs in the matrix and σ_{fc}^{b} is the flexural strength of the MWCNTs reinforced concrete. The MWCNTs must be embedded in the matrix to some critical length (L_c^{crit}) so that the inclusion may observe full tensile capacity. The L_c^{crit} may be termed as critical pull-out length which may be evaluated from following expression [44]:

$$L_c^{crit} = L_c \sqrt{\sigma_c^{\mu} \eta_o V_c} \, / \, \sqrt{2\sigma_{fc}^{\mu}} \tag{3}$$

The results of the above expression indicate that the critical pullout length of MWCNTs increases with their percentage inclusion in the concrete (Fig. 6). The increase in critical length reduces the effective stress that may be developed in the MWCNT bridging a growing crack. Fig. 7 represents the stress development in MWCNTs as a relation of their embedment lengths.

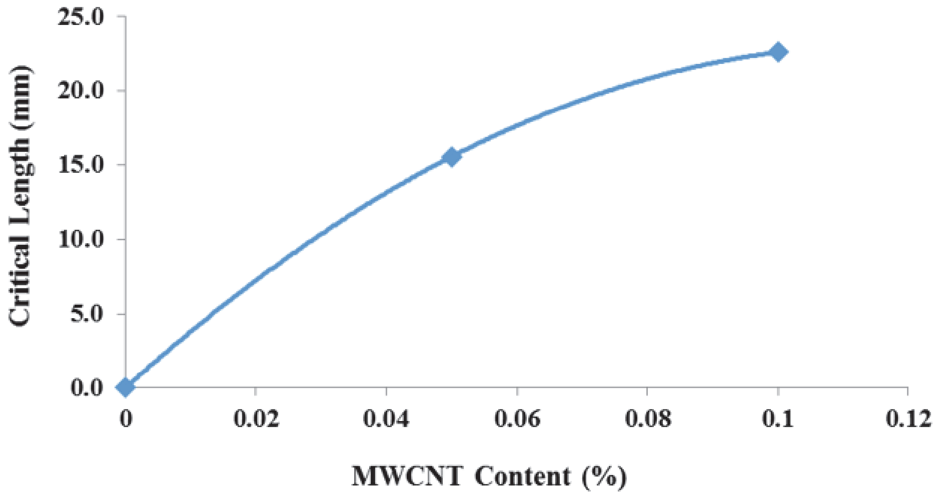

Figure 6: Relationship of critical pullout length and content of MWCNTs in the concrete matrix.

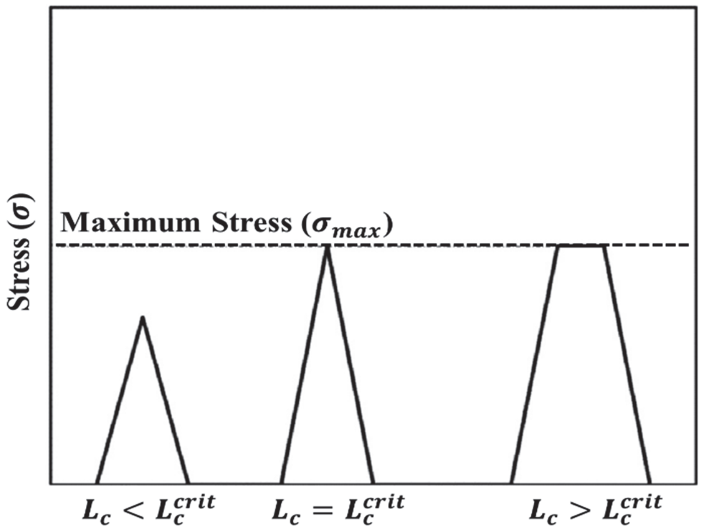

Figure 7: Relationship of embedment length and stress development in MWCNTs [44]

Conclusions

In this paper, the remarkable improvement in the mechanical properties of concrete was observed on the inclusion of small fractions (i.e. 0.05 wt% of cement) of MWCNTs in concrete matrix. The effects and behavior of MWCNTs addition is purely dependent upon the dispersion and content of MWCNTs in the mix. Many other factors other than dispersion, are also associated with effective outcomes of the inclusion of MWCNTs in concrete i.e., size, aspect ratio, purity etc. of MWCNTs being utilized in the mix. From the present study, it may be concluded that the same amount of MWCNTs may exhibit different behavior in certain mechanical properties of concrete mix i.e. the lower amount of MWCNTs is fruitful in case of enhancement of tensile and flexural strength as evident by the theoretical analysis but in case of compressive strength the larger fraction is more effective.

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