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Modified arcan tests for concrete with multi-walled carbon nanotubes

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ABSTRACT

An experimental study consisting 70 tests have been conducted to study the influence of addition of reinforcing fibers on concrete specimens. The experimental program included concrete specimens that were tested with modified Arcan test machine with different notch lengths. The reinforcing effect of highly dispersed multi-walled carbon nanotubes (MWCNTs) in concrete has been investigated. The results revealed that inclusion of CNTs in the design mix improve both the tensile fracture characteristics and compressive strength when not mixed with a surfactant compound. The improvement in the mechanical properties specimens with the addition of CNTs are observed more clearly with increasing curing age. The mixing process to achieve uniformly dispersed and properly mixed mortar however requires specialized equipment, such as ultrasonic mixers. The results also indicated some dependency on the size of the specimens, which is a well known phenomenon that is observed for brittle heterogenous materials such as concrete. Copyright © 2014 VBRI press.

Keywords: Arcan test; notch effect; carbon nanotubes; mechanical properties.

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Introduction

The present study is valuable for ensuring a safe and effective utilization for concrete structures. Size effect is one of the most important practical subject of fracture mechanics as well as one of the easiest to validate by experimental testing by failure approaches **[1]**. Understanding the size effect is essential for structural materials. The size effect can also be exploited by the measurements of tension fracture parameters.

CNTs present several distinct advantages as reinforcing materials for high strength/performance cementitious composites as compared to more traditional fibers. Firstly, they exhibit significantly greater strength and stiffness than conventional fibers **[2]**. Secondly, because of their high length-to-diameter aspect ratio they intercept with the nanocracks and demand significantly higher energy for crack propagation. Lastly, provided that CNTs are evenly dispersed, fiber spacing is uniformly distributed in the mix which prevents local failure modes with significant strength gain **[3]**.

Few attempts by researchers have been made to add CNTs as reinforcement in cementitious matrices. Makar et al [4-5] investigated the reinforcing effect of 2.0 wt.% CNTs in cement using ultra high resolution electronic microscope (SEM) and Vickers hardness measurements.

The results indicated that CNTs may affect the early hydration progress, producing higher hydration rates. Li et al. **[6, 7]** employed a carboxylation procedure to improve the bonding between 0.5 wt.% MWCNTs and cement matrix and obtained a 25% increase in flexural strength and a 19% increase in compressive strength. Saez de Ibarra et al. **[8]** measured the stiffness of cement samples reinforced with MWCNTs and singlewall carbon nanotubes (SWCNTs) using an atomic force microscopy (AFM) nanoindentation technique and reported modest gains in the Young's modulus. Cwirzen et al. **[9-10]** investigated the mechanical properties of cement matrices reinforced with different concentrations of MWCNTs.

The results showed no increase in the flexural strength but a slight increase in compressive strength of the cement paste with the addition of CNTs. Researchs **[11-15]** on the reinforcing effect of MWCNTs in cement matrix (water/cement=0.5) indicated that CNTs can strongly reinforce the cement paste matrix by increasing the flexural strength and the Young's modulus of plain cement paste by 25% and 50%, respectively. It is also shown that for proper dispersion the application of ultrasonic energy is strictly necessary and for complete dispersion.

A total of 70 Modified Arcan tests **[16]** were performed to investigate the mixed-mode fracture behavior and parameters of specimens. Two type of specimens were used in the Modified Arcan test program; the first group was 35 plain mortar specimens, and the second was 35 mortar specimens with CNTs. Additionally, 54 compression tests of cylinders with different diameters were conducted to study the compressive strength influence of including CNTs in the mortar. Similarly, half (27) of the compression test specimens were with CNTs.

Cracking of concrete is a big problem for the safety of concrete structures. Since concrete tension strength is much lower compared to its compression strength, it is vital to improve the tension strength. But the experimental results on the cement paste with carbon nano tube cannot truly represent the real fracture properties of concrete. Accordingly, the specimens for this study were cast CNTs with mortar. So the research on the fracture behavior of concrete with CNTs is of important.

Recent cement based material studies have mostly focused on developing high-performance cementitious composites, which exhibit high compressive strengths. Such composites however, exhibit also extremely brittle failure, low tensile capacity and appear to be sensitive to early age microcracking as a result of volumetric changes due to high autogenous shrinkage stresses. To overcome the disadvantages of the cement paste, it is recommended to use reinforcement cementitious materials which are typically provided at micro scale using carbon nanotubes (CNTs).

This research work has the significance that, to the best of the authors' knowledge, this study is the first to study mechanical properties of mortar with CNTs. The effect of CNTs for specimens subjected to mixed mode, tension (Mode I) and shear (Mode II), loadings has been studied using a unique test setup. The gained understanding of the effect of CNTs in tension is needed for improving the resistance of concrete structures to earthquakes.

Experimental

Materials

The concrete mix proportions of *w/c/s/g/SP* =0.5:1:2.81:1.81:0.03, mortar with CNTs was CNTs/*c*=0.005 and surfactant was Surf./CNTs=1.5 (by weight). In this mix ratio, *w* stands for water, *c* for cement, *s* for sand, *g* for gravel, *SP* for high performance superplasticiser Glenium 51 (Basf Chemical Company) based on modified polycarboxylic ether. All of the specimens were cast from the same batch of concrete in order to minimize statistical scatter of the results. Portland cement with additive (KPC42.5, nominal compressive cylinder strength of 42.5MPa at 28 days), and Elmadağ (a town near Ankara) region crushed aggregate was used as sand and gravel. The maximum aggregate sizes for all of the beams was d_a=6mm. The definiton of "gravel" is used for diameters between 6 and 2mm, and the definition of sand is used for diameters less than 2mm. During the sieving process a 4mm sieve was used to divide the aggregate into two seperate batches, one with diameter between 6 to 4mm coarse gravel, and the other between 4 to 2mm for fine gravel. Each batch was combined equally in weight to compose the coarse and fine gravel as a part of the admixture.

In this test one kind of multiwall carbon nanotube was used (MWNTs). Properties of CNTs are given in **Table 1**. As seen the length-to-diameter aspect ratio of the nano tubes is beyond 1000. The purity percentage of carbon nano tube is 90% as weight. Test results suggest that MWCNTs improve the nano- and macromechanical properties of concrete.

Table 1. Properties of CNTs.

Outer Diameter	>50nm
Length	10-20mm
Purity	>90wt%
Ash	<1.5wt%
Specific Surface Area	>40m^2/g
Electrical Conductivity	>100s/cm

Superplasticier (SP) was used to reduce the water content in the mortar mix. Modified polycarboxylic ether based Glenium 51 was used as a superplasticier with a constant content ratio of 0.03 by weight of the cement.

Surfactant was also used for some of the specimens. The compound was an anionic surfactant used in the mix and made of sodium dodecyl sulfate. The surfactant is an organic compound with the formula $CH_3(CH_2)_{11}OSO_3Na$.

Test specimens

Behaviors of 70 notched specimens with different notch length were investigated by subjecting to mixed-mode loading. The tests were conducted at the Structural Mechanics Laboratory of Gazi University, Ankara, Turkey. The prism specimens had constant rectangular cross section with sizes, 180x120x20mm as delineated in **Fig. 1**.

All specimens tested in Arcan test machine were specimens with edge notch. Various notch depths are considered to determine the size effect and fracture mechanics parameters. For these tests, the notch ratios were chosen as α=0.16, 0.25, 0.32, 0.48, 0.64 where α=a/d. In this formula, *a* is length of notch, *d* is the depth of specimen.

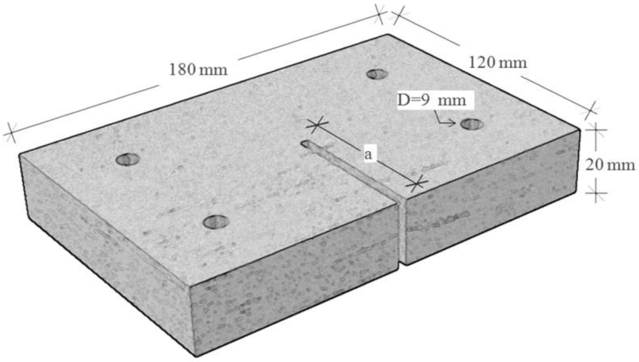

Fig. 1. Concrete test specimen.

For the preparation of the CNT nanocomposites ordinary Portland cement and MWCNTs were used. Prior to their addition to the cement MWCNTs were dispersed in water by applying ultrasonic energy. Also prisms did not contain surfactant for age 45days, for other ages prisms were casted with surfactant.

A 750W cup-horn high intensity ultrasonic processor (Sonics VCX 750 vibra cell) was used to apply constant energy (1900-2100 J/min) to the CNT dispersions. Ultrasonic processor was used as a 3 minutes with 38% amplitude. After the sonication, cement was added into the CNT dispersions at a water to cement ratio of 0.50. The percentage of water present in the slurry was considered so as to maintain water to binder ratio of 0.50. After that sand, aggregate were added to slurry. Following the mixing procedure, the mortar was cast in the molds. After demolding, the specimens were cured in water saturated with lime until the test day.

Fig. 2. Specimens after notching.

The specimens were tested in three different ages. First age was 45 days and no surfactant used in the mix, the second age was 37 days with surfactant, the third age was 29 days with surfactant. The ages 45 and 37 days were tested under the slope of notch of φ=30°, age 29 was tested under φ=45°. Notches are made 3 days before the testing by a circular sawmachine. Notched specimens are shown in **Fig. 2** where, a is the notch length, d is the depth of prisms was same and equal to 120mm for all the specimens.

The tests were conducted using Shimadzu AG-X series axial test frame with a maximum load capacity of 100 kN. The tests were carried out under a constant stroke rate that varies for specimen with different notch length. The stroke rate was selected to achieve the maximum load for each specimen in approximately 4 minutes. The stroke rate was 0.2mm/min for specimens with α=0.64, 0.3 mm/min for α=0.32 and 0.4 mm/min for α=0.16, 0.21, 0.25.

The modified Arcan tests were carried out to determine the fracture behavior. The experimental set up is shown in **Fig. 3**, where the Arcan test apparatus connecting the specimen to test machine was made of aluminum. The modified test frame for Arcan test allows subjecting specimens to a combination of Mode I (tension) and Mode II (shear) loading. In this study, Mode I and Mode II type failures were studied by subjecting the specimens to pulling angles of φ=30° and 45°.

Fig. 3. Experimental setup

The specimen name *P* stands for notched prisms and follows with the designated number of the specimen. Five different notch depth ratio α=a/d was used as α=0.16, α=0.25, α=0.32, α=0.48 and α=0.64. The smallest notch length was kept longer than the total pin diameter, 2x9=18mm therefore the smallest notch length is choosen as 19mm to prevent failure around pin region.

A total of 54 cylinder compression tests were performed with three different size cylinders of 75mm, 37.5mm, and 18.75mm in diameter and 150mm, 75mm, and 37.5mm in length, as shown in **Fig. 4**. The cylinder testing was done parallel to the Arcan test for each cast from the same batch both with and without CNTs.

Results

The test results of the specimens are presented in **Table 2**. As explained earlier, although special caution was given on the minimum notch length some of the specimens failed at the pin region, such as specimen P4 for plain prisms. The goal of the experimental program was to test at least three specimens for each different notch-to-depth (α) ratios. However, the pin failures led to testing less than the desired number of specimens; as in the case of having fewer successful tests for specimens with α less than 0.25. Although some of the specimens have failed from the pinned ends, most of them failed from the expected notched part of the specimen. A total of 70 specimens are tested with 35 of them including CNTs.

Fig. 4. Some of the specimens during the test.

Table 2. Test results of prisms with and without CNTs.

Name	φ (^{0})	*a=a/D*	*Age* (day)	P_0 (kN)	P_{c0} (kN)	Surf /CNT
P1	30	0.16	45	1.81	2.21	---
P2	30	0.21	45	1.26	2.16	---
P3	30	0.21	45	2.05	2.17	---
P4	30	0.25	45	---	2.09	---
P5	30	0.32	45	1.62	2.09	---
P6	30	0.32	45	2.08	1.60	---
P7	30	0.32	45	1.54	1.71	---
P8	30	0.32	45	0.84	1.59	---
P9	30	0.64	45	0.39	0.80	---
P10	30	0.64	45	0.63	0.43	---
P11	30	0.64	45	0.66	0.66	---
P12	30	0.64	45	---	0.71	---
P1	30	0.21	37	2.45	1.43	1.5
P2	30	0.25	37	1.70	1.25	1.5
P3	30	0.25	37	2.06	0.76	1.5
P4	30	0.25	37	2.14	1.27	1.5
P5	30	0.32	37	1.94	0.91	1.5
P6	30	0.32	37	1.55	1.08	1.5
P7	30	0.32	37	1.47	1.23	1.5
P8	30	0.32	37	1.66	0.97	1.5
P9	30	0.64	37	0.56	0.31	1.5
P10	30	0.64	37	0.56	0.21	1.5
P11	30	0.64	37	0.58	0.29	1.5
P12	30	0.64	37	0.74	0.44	1.5
P1	45	0.25	29	1.67	0.66	1.5
P2	45	0.25	29	2.02	0.84	1.5
P3	45	0.25	29	1.87	1.22	1.5
P4	45	0.25	29	1.58	---	1.5
P5	45	0.32	29	1.57	0.56	1.5
P6	45	0.32	29	1.69	0.61	1.5
P7	45	0.32	29	1.41	0.73	1.5
P8	45	0.32	29	---	0.93	1.5
P9	45	0.64	29	0.75	0.21	1.5
P10	45	0.64	29	0.44	0.29	1.5
P11	45	0.64	29	0.52	0.16	1.5

During the tests; failure loads and failure displacements are recorded. The failure displacement is measured as the total displacement of the test specimen (the test setup is considered to be rigid). In this study only the failure loads are considered in understanding the behavior and are reported in tables. The specimens with different notch length-to-depth ratio were tested in one day at the age for 45 days, 37 days and 29 days. Photos of some of the specimens (with different notch length-to-depth ratios) during the test are given in **Fig. 5**.

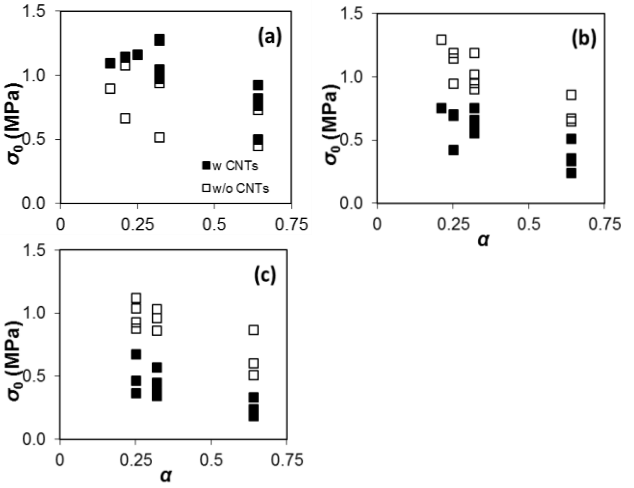

Fig. 5. Effect of ratio of Notch length to width at different ages; (a) 45 days, (b) 37 days and (c) 29 days.

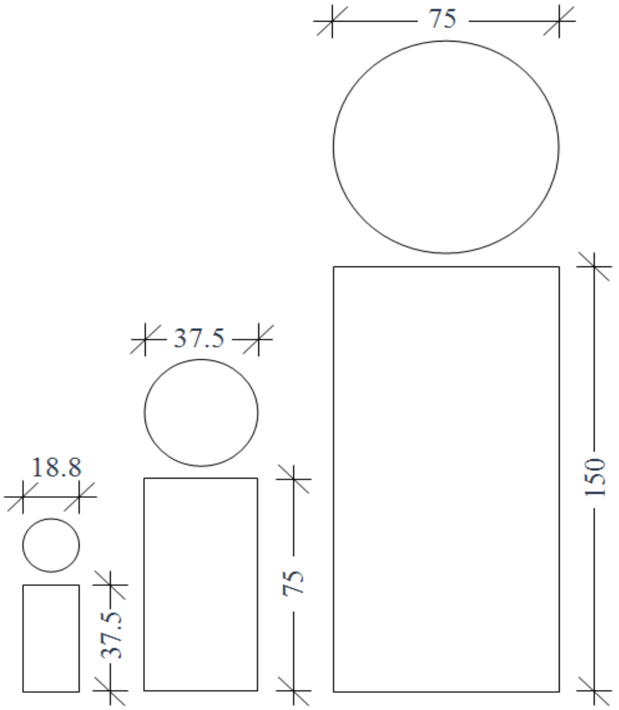

Fig. 6. Schematic view of test cylinders.

The effect of notch length to failure stress is given in **Fig. 6**. In this figure horizontal axis is the notch depth ratio α, vertical axis is the axial stress calculated at the critical section from Eq. 1 by using the net cross-sectional area of the specimens.

$$\sigma = P_0 \cos\varphi / [(d-a)b] \quad (1)$$

P_0 is the maximum (failure) load of specimens taken from Table 2. In this equation; φ is the loading angle, α is the notch length-to-depth ratio, Age is the specimen age from the day of casting. As seen in **Fig. 6**, tensile strength is decreasing nonlinearly with increasing notch length ratio. In this one dimensional similarity tension stress was decreasing sharply, means strong size effect exists.

Mortar specimens with CNTs maximum load of prisms were given as P_{c0}. Similarly, for these specimens Eq. 1 were used for stress calculation. In **Fig. 5** filled square markers shows with CNTs prisms results, empty square markers shows without CNTs prisms results.

Test results of cylinders with and without CNTs specimens were given in **Table 3**. For three different ages cylinders were tested at 46, 38 and 30 days. In this table name of cylinders C stands for cylinders and the following number gives assigned specimen number. Failure loads of cylinders were given as P0 for without CNTs, P_{c0} for with CNTs.

Table 3. Test results of cylinders with and without CNTs.

Name	*l* (mm)	*d* (mm)	*Age* (day)	P_0 (kN)	P_{c0} (kN)
C1	150	75	46	92.11	106.55
C2	150	75	46	---*	---
C3	150	75	46	---	---
C4	75	37.5	46	19.72	33.74
C5	75	37.5	46	31.30	42.87
C6	75	37.5	46	38.58	42.81
C7	37.5	18.8	46	8.55	10.01
C8	37.5	18.8	46	6.78	7.26
C9	37.5	18.8	46	5.32	8.23
C1	150	75	38	116.73	90.74
C2	150	75	38	---	102.04
C3	150	75	38	---	---
C4	75	37.5	38	---	---
C5	75	37.5	38	25.59	23.76
C6	75	37.5	38	19.57	23.39
C7	37.5	18.8	38	8.55	3.57
C8	37.5	18.8	38	7.10	7.91
C9	37.5	18.8	38	8.07	5.33
C1	150	75	30	104.79	84.44
C2	150	75	30	---	94.44
C3	150	75	30	---	---
C4	75	37.5	30	6.40	8.31
C5	75	37.5	30	24.36	13.14
C6	75	37.5	30	13.47	12.25
C7	37.5	18.8	30	6.62	5.02
C8	37.5	18.8	30	11.14	4.03
C9	37.5	18.8	30	8.55	5.81

* missing cylinders

Test results are given in **Fig. 7** for different age cylinders with and without CNTs. Vertical axis show that stress was obtained according to Eq. 2.

$$\sigma = P_0 / (\pi d^2/4) \quad (2)$$

(Eq. 1)

Horizontal axis is the cylinder diameter *d*. Effect of CNTs are similar to the observations noted for the Arcan tests, where the mix not containing the surfactant solution produces improved strength.

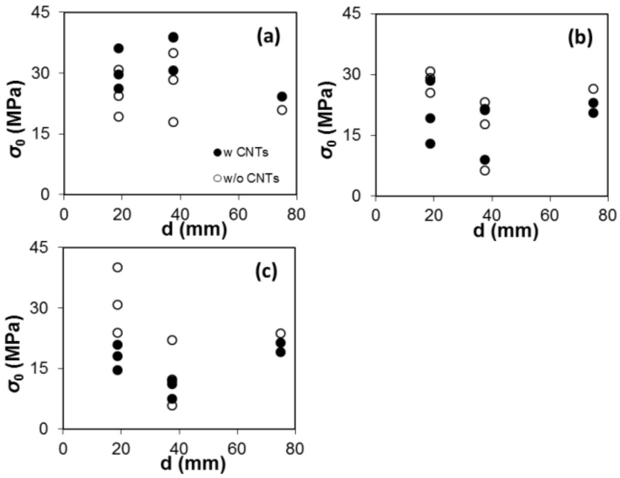

Fig. 7. Failure strength of cylinders at different ages; (a) 46 days, (b) 38 days and (c) 30 days.

Discussion

Ultrasonication is a common physical technique used to disperse CNTs into base fluid. Ultrasonic processors convert line voltage to mechanical vibration. These mechanical vibrations are transferred into the liquid by the probe creating pressure waves. This action causes the formation and violent collapse of microscopic bubbles. This phenomenon, reffered to as cavitation, creates millions of shock waves, resulting in dispersion of objects and surfaces within the cavitation field. For proper dispersion of the CNTs, application of ultrasonic energy is absolutely required.

Some researchers **[14, 15]** have reported that inclusion of surfactant is necessary to increase strength of the mix when used with CNTs. However, in this study the specimens that included surfactant in the mix design resulted in even lower strength than plain concrete mixes. This may have resulted from using a different type of surfactant which precluded the strength advantages that would be obtained from CNTs.

Conclusions

Mechanical properties of the mortar reinforced with MWCNTs have been investigated. According to the test results, there is significant strength gain in the specimens that include CNTs without surfactant. Both the tension and compressive strength have risen for these specimens. For the specimens tested using the Arcan test apparatus, some size effect was observed where strength of notched specimens decreased nonlinearly with the increasing notch length ratio. The fracture mechanics test results indicates that the fracture properties of micro concrete and mortar are increased through proper dispersion of very low amount of CNTs (CNTs/c=0.005). Dispersing carbon nanotubes in micro concrete and mortar is achieved by applying ultrasonic energy. Affect of CNTs gain more strength with increasing age of the specimens.

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