

Fracture mechanics characterization of mortar nanocomposites reinforced with carbon nanotubes

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The objective of this paper is to provide a fracture mechanics characterization of mortar nanocomposites reinforced with multiwall carbon nanotubes (MWCNTs). The critical stress intensity factor, K_{Ic} , the strain energy release rate, G_{Ic} , the effective crack length, a_c , and the crack tip opening displacement, $CTOD_c$ are determined using three-point bend specimens. It was established that reinforcement of mortar with MWCNTs provides excellent improvement in the above fracture mechanics quantities.

Keywords: fracture mechanics, multi-wall carbon nanotubes, stress intensity factor, energy release rate, crack tip opening displacement, mortar nanocomposites, cementitious materials.

1. Introduction. Defects are of utmost importance in the mechanical and failure behavior of cementitious materials, like cement pastes, mortars, concrete, etc. These materials are heterogeneous with complex microstructure and can be modeled at various scale levels including the nano, micro, meso and macro levels. A simple way of modeling cementitious materials is to consider them as a two-phase particulate composite. In cement pastes the matrix is the hydrated cement gels and the reinforcement is the unhydrated cement particles. In mortar the matrix is the cement paste and the reinforcement is the fine aggregates. In concrete the matrix is the mortar and the reinforcement is the coarse aggregates.

Failure of cementitious materials is the result of initiation, growth and coalescence of defects, like pores, cracks, etc. starting from the nano, and progressing to micro, meso and macro scale levels. For example, in concrete microcracks are usually present, even before loading, at regions of high material porosity near the interfaces between the coarse aggregates and the mortar. Cracks may also be present in mortar. When the structure is subjected to applied loads, temperature difference, etc., both types of cracks start to increase and new cracks are formed. The interface cracks propagate into the mortar and are connected with the mortar cracks. When a sufficient number of microcracks coalesce a macrocrack is formed. The damage zone ahead of a traction free crack referred to as fracture process zone (FPZ) plays an important role in the analysis of growth of cracks.

Within the FPZ many failure mechanisms including microcracking, debonding of cement-matrix interface, crack deviation and branching take place. These mechanisms contribute to the energy of fracture. Carbon nanotubes (CNTs) with their supreme stiffness, high strength and aspect ratio are excellent reinforcing candidate materials, offering unprecedented improvement in both strength and stiffness [1, 2].

Attempts have been made to stop or delay the growth of cracks at micro and macro scale levels by reinforcing cementitious materials by fibers at these levels [3]. Microfibers are defined as fibers with diameters less than 50 μm and macrofibers with diameters less than 500 μm . Microfibers delay the process by which microcracks develop to form macrocracks by bridging the cracks and transferring the load, while macrofibers improve the post peak toughness of the material. Cracks, however, are formed at the nano scale level. To suppress the initiation and growth of these cracks, the use of nano fibers was introduced.

The superior mechanical, electrical and chemical properties of carbon nanotubes (CNTs) make them candidate materials for nano reinforcement. These materials, with aspect ratio greater than 1000 have Young's modulus around 1 TPa, tensile strength of 65–93 GPa, and maximum strain of 10–15%. CNTs form agglomerates or bundles and adhere together with Van der Waals forces which makes it particularly difficult to separate. Early attempts to add CNTs to cementitious materials have failed due to poor adhesion [4–10].

Konsta – Gdoutos and coworkers [1, 11–13] developed a revolutionary method for effective dispersion of CNTs in cement pastes, mortars and concrete. The dispersion of different lengths of multi-wall carbon nanotubes (MWCNs) in water was achieved by applying ultrasonic energy and using a surfactant. They studied the effects of ultrasonic energy and surfactant concentration on the dispersion of CNTs and concluded that an optimum of weight ratio of surfactant to CNTs should exist. They found that small amounts of effectively dispersed MWCNs (0.025–0.08 wt % of cement) increase the flexural strength of cementitious composites. Furthermore, they studied the effect of carbon nanofibers (CNFs) and showed that the nanocomposites exhibit superior mechanical and electromechanical properties. They demonstrated that CNFs and carbon fibers (CFs) can be used in concrete to successfully create a conductive network for stress/damage detection. They found that in cement nanocomposites reinforced with MWCNs the modulus of elasticity increases by 45% with respect to the cement paste material, while the flexural strength increases by 25% for a reinforcement of 0.08 wt %. Moreover, they established that longer MWCNTs are more effective than shorter ones.

In the above works the effect of MWCNTs and CNTs on the Young's modulus, strength and ductility of cementitious material was studied. However, fracture mechanics characterization of cementitious material requires determination of critical fracture mechanics quantities including stress intensity factor, K_I , critical strain energy release rate, G_I , critical crack mouth opening displacement, $CMOD_c$ and critical crack length, a_c . Failure by crack propagation can be described when stress intensity factor, strain energy release rate or crack mouth opening displacement reach their critical values. These quantities are functions of applied loads and the size and dimensions of the structure, while their critical values are material properties.

The foundations of the application of fracture mechanics to concrete were laid down by the pioneering work of Hillerborg [14, 15] who introduced the fictitious crack model of concrete, in an analogous way to the Dugdale – Barenblatt model of metals. After that, the development of the field was explosive and the theory appears to be matured for

applicability to design. Early works on the application of fracture mechanics to concrete are listed in references [16–27]. Reda et al. [28] used the effective crack model developed by Karihaloo and Nallathambi [29] to determine the critical energy release rate for high performance concrete, mortar, fiber reinforced concrete and masonry units. Moukwa et al. [30] studied the effect of alumino-silicate clays on the critical stress intensity factor and critical crack tip opening displacement. They concluded that appropriate use of silica fume and alumino-silicate clays can increase the ductility and strength of cementitious materials. Das et al. [31] used notched three-point bend specimens to determine the critical stress intensity factor and critical crack tip opening displacement in mortars in which the Ordinary Portland Cement (OPC) was replaced by limestone or a combination of limestone and fly ash/metakaolin. The fracture quantities were determined by using a two-parameter method and a non-contact digital image correlation. It was obtained that blends of OPC replacement materials and limestone can lead to enhanced fracture mechanics properties and ductility. Sarker et al. [32] studied the fracture characteristics of geopolymer (GPC) and OPC concrete using three-point bend specimens. They found that the critical stress intensity factor is higher for the GPC than the OPC concrete. Nikbin et al. [33] studied the fracture characteristics of self-compacting concrete using notched three-point bend specimens. Stynoski et al. [34] studied the fracture properties of various Portland cement mixtures containing silica fume, carbon nanotubes and carbon nanofibers using notched three-point bend specimens and the two-parameter method of Jenq and Shah [2]. They found an improved fracture mechanics performance of mixtures containing carbon nanotubes and carbon nanofibers.

It is the objective of the present work to characterize the fracture mechanics behavior of cementitious materials reinforced with well-dispersed carbon nanotubes. Experiments were performed on three-point bending precracked specimens of cement pastes without and with MWCNTs reinforcement. The excellent reinforcing capabilities of MWCNTs are demonstrated by a significant improvement in critical stress intensity factor (128.6%), critical strain energy release rate (154.9%), critical crack tip opening displacement (39.7%) and effective crack length (10.3%), resulting in advanced materials for civil engineering applications.

2. Fracture mechanics of cementitious materials. Fracture mechanics characterization of cementitious materials, like cement pastes, mortars and concretes is important for various reasons. First, it is generally accepted that material separation is better described by energy principles rather than by stress or strain. During fracture, new material surfaces are created. The energy required to create new material surfaces during the fracture process is a fundamental characteristic quantity of the material. Application of fracture mechanics gives a basis for the understanding of the fracture process. Second, high strength concrete with compressive strength higher than 100 N/mm^2 presents extremely brittle behavior, and fracture mechanics is well suited for studying its failure. Third, design of large structures, like dams, nuclear reactors, behave in a rather brittle manner, and their design will be benefited tremendously by fracture mechanics. Fourth, characterization of fracture toughness by the area under the stress-strain curve of the material cannot be used to quantify fracture toughness, since it is size and geometry dependent. Fifth, classical theories of fracture based on stress or strain cannot explain the size effect, according to which the ultimate stress of geometrically similar structures of different sizes depends on the size of the structure. The size effect can only be explained by fracture mechanics.

Direct application of fracture mechanics to cementitious materials is questionable, since the values of the critical strain energy release rate are specimen size dependent and, therefore, they could not be used as a characteristic material property. It was realized that cementitious materials require a different kind of fracture mechanics than metals. In both metal and concrete structures, nonlinear zones of small (small scale yielding approximation treated by linear elastic fracture mechanics) or normal sizes (ductile fracture) develop at the crack tip. However, in ductile/brittle metals the material in the nonlinear fracture process zone ahead of the crack tip undergoes hardening or perfect plasticity, whereas in concrete the material undergoes softening damage. In cementitious materials within the fracture process zone (FPZ) many micro-failure mechanisms including matrix microcracking, debonding of cement-matrix interface, crack deviation and branching take place. All these mechanisms contribute to the energy of fracture. The size of the fracture process zone ahead of the stress-free crack depends on the geometry and size of the structure and the type of material. For cement paste the length of the FPZ is of the order of a millimeter, for mortar is about 30 mm, for normal concrete of coarse-grained rock is up to 500 mm, for dam concrete with extra-large aggregates is about 3 m, for a grouted soil mass is around 10 m and in a mountain and jointed rock values of 50 m may be typical. On the other hand the length of the FPZ in a fine-grained silicon oxide ceramic is of the order of 0.1 mm, and in a silicon wafer of the order of 10–100 nm.

3. Experimental determination of K_{Ic} , G_{Ic} and $CMOD_c$. **3.1. General remarks.** Direct application of fracture mechanics principles have been used for the experimental determination of the critical fracture toughness of cementitious materials. The methodology has been similar to the experimental determination of the critical strain energy release rate, G_{Ic} , or stress intensity factor, K_{Ic} , in metals. Notched three-point bend specimens have been most popular. The specimens were loaded to a progressively increasing load and the load versus deflection of crack tip opening displacement response was recorded. The value of G_{Ic} , or K_{Ic} , is determined from the peak load or the load at the intersection with the secant of slope 95 % of the initial slope with the load-displacement curve. The values of G_{Ic} , or K_{Ic} obtained were dependent on the size of the specimen and its geometrical configuration. Thus direct application of linear elastic fracture mechanics for the characterization of the fracture behavior of cementitious materials is not successful.

3.2. The Jenq – Shah method. In an effort to provide reliable critical fracture toughness values of cementitious materials, Jenq and Shah [2] proposed a methodology based on the compliance of the notched specimen. The basic idea behind the method is to determine an effective, not the initial, crack length. This effective crack length takes into consideration the inelastic phenomena that take place in the fracture process zone ahead of the crack tip. The effective crack length is equal to the actual crack length plus the length of the fracture process zone. For the application of the method the relationship between the compliance, defined as the value of the crack tip opening displacement per unit load, and the crack length for special specimen types (three-point notched bend specimen) is established. The unloading compliance of the specimen is determined by unloading the specimen after reaching the peak load. The effective crack length is calculated from the relationship between the compliance and the crack length using the unloading compliance. The critical value of strain energy release rate, G_{Ic} , or stress intensity factor, K_{Ic} , is calculated from the peak load and the effective crack length using linear elastic fracture mechanics formulas. It has been established that K_{Ic}

determined in this way is independent of the specimen type and size. Thus, determination of K_{Ic} needs two quantities measured from the test, the unloading compliance and the peak load. Simultaneously with K_{Ic} the modulus of elasticity and the critical value of the crack tip opening displacement are determined.

The experimental determination of the above quantities is based on the load versus crack mouth opening displacement (CMOD) curve for a loading-unloading cycle of a three-point bend specimen (Fig. 1).

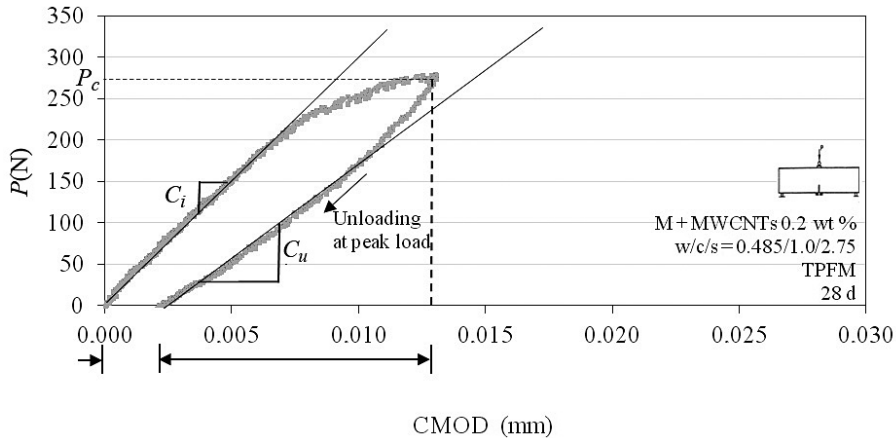


Fig. 1. Typical load-CMOD curve from loading and unloading procedure for 28 d mortar reinforced with well dispersed MWCNTs at an amount of 0.2 wt % of cement.

From the curve the following quantities are measured: the compliances for the loading and unloading parts and the maximum load.

The modulus of elasticity, E , is calculated by

$$E = \frac{6Sa_0g_2(\alpha_c)}{C_i b^2 t}, \quad (1)$$

where C_i – the compliance of the loading part of the load-CMOD curve, $\alpha_c = (a_0 + HO)/(b + HO)$, a_0 is the crack length, S is the span length, b is the specimen depth, t is the specimen thickness, HO is the length defined in Fig. 2, between the two mark points on the bottom of the beam, $g_2(\alpha_0)$ – geometric function defined by

$$g_2(\alpha_0) = 0.76 - 2.28\alpha_0 + 3.87\alpha_0^2 - 2.04\alpha_0^3 + \frac{0.66}{(1 - \alpha_0)^2}. \quad (2)$$

In a similar way, the modulus of elasticity E is calculated from the compliance of the unloading part of the load-CMOD curve as

$$E = \frac{6Sa_c g_2(\alpha_c)}{C_u b^2 t}, \quad (3)$$

where C_u is the compliance of the unloading part of the load-CMOD curve, a_c is the effective crack length, $\alpha_c = (a_c + HO)/(b + HO)$.

The unloading compliance is taken within 95 % of the peak load calculated from the load-CMOD curve. The value of the effective crack length a_c is calculated by equating

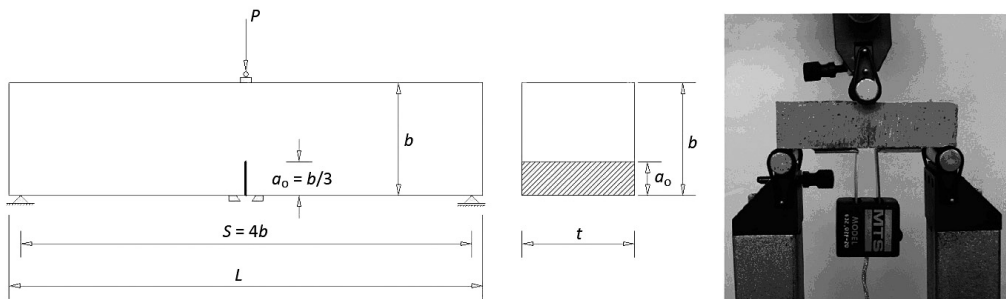


Fig. 2. Experimental setup for the TPFM testing.

the values of the modulus of elasticity E defined from Eqs (1) and (3). This results in the following equation

$$a_c = a_0 \frac{C_u g_2(\alpha_0)}{C_i g_2(\alpha_c)}. \quad (4)$$

Equation (4) is solved numerically for the determination of the critical crack length a_c .

The critical stress intensity factor is calculated by the equation

$$K_{IC}^S = 3(P_{cr} + W_h) \frac{S \sqrt{\pi a_c} g_1(a_c/b)}{2b^2 t}, \quad (5)$$

where P_{cr} is the peak load, $W_h = W_{ho} S/L$, W_{ho} is the self weight of the beam, and

$$g_1\left(\frac{a_c}{b}\right) = \frac{1.99 - (a_c/b)(1 - a_c/b)[2.15 - 3.93(a_c/b) + 2.70(a_c/b)^2]}{\sqrt{\pi}(1 + 2a_c/b)(1 - a_c/b)^{3/2}}. \quad (6)$$

The critical crack tip opening displacement is calculated by

$$CTOD_c = \frac{6(P_c + 0.5W_h)S a_c g_2(a_c/b)}{E b^2 t} [(1 - \beta_0)^2 + (1.081 - 1.149(a_c/b))(\beta_0 - \beta_0^2)]^{1/2}, \quad (7)$$

where $\beta_0 = a_0/a_c$, $g_2(a_c/b)$ is calculated from Eq. (2) with $\alpha_0 = a_c/b$.

Based on the values of K_{IC}^s and $CMOD_c$ Jenq and Shah [2] introduced a material length Q by

$$Q = \left(\frac{E C T O D_c}{K_{IC}^s} \right)^2. \quad (8)$$

The material length Q can be used to characterize the brittleness of the material. The smaller the value of Q and more brittle the material is. It was found that values of Q are in the range of 12.5–50 mm for hardened cement paste, 50–150 for mortar, and 150–350 for concrete.

4. Experimental work. 4.1. Materials and specimens. The material investigated in this work was a cement-based composite with the matrix reinforced by multi-walled carbon nanotubes (MWCNTs). The nanotube reinforcement was of 10–100 μm length and 20–40 nm outer diameter. Characteristic properties of MWCNTs and are shown in Table 1.

Type I ordinary Portland cement (OPC) and standard sand according to EN 196–1 were used for casting the cement paste and mortar specimens. To disperse the MWCNTs

Table 1. Properties of MWCNTs

Nanotubes	Diameter, nm	Length, μm	Surface area, m^2/gr	Bulk density, gr/cm^3	Aspect ratio
MWCNTs	20–45	≥ 10	> 200	≤ 0.18	500

homogeneously in the mixing water, MWCNT suspensions were prepared by adding the MWCNTs in an aqueous surfactant solution that includes a commercially available polycarboxylate based surfactant (SFC). The resulting dispersions were sonicated at room temperature following the method described by Konsta-Gdoutos et al. [11, 12]. A constant surfactant to MWCNTs weight ratio of 4.0 was found to achieve effective dispersion. Ultrasonic energy is applied to the aqueous samples by a 500 W cup-horn high intensity ultrasonic processor with a standard probe of a diameter of 13 mm and temperature controller. The sonicator is operated at amplitude of 50 % so as to deliver constant energy rate of 1900–2100 J/min, at cycles of 20 seconds in order to prevent overheating of the suspensions.

After completion of the sonication procedure, the MWCNT suspensions were added into the OPC and sand at a constant water to cement ratio $w/c = 0.485$ and sand to cement ratio $s/c = 2.75$. Mixing of the materials was performed according to procedure outlined by ASTM 305 using a standard robust mixer capable of operating from 140 ± 5 to 285 ± 10 revolutions per minute (r/min). After mixing, one set of the mixture was cast in $20 \times 20 \times 80$ mm oiled molds. Following demolding, the samples were cured in lime-saturated water until testing. A 6 mm notch was introduced into the prismatic $20 \times 20 \times 80$ mm specimens using a water-cooled band saw machine.

The same procedure was used for both cement pastes and mortars. Values of an estimated number of nanotubes per unit volume of the cementitious matrix are also included in the Table 2, expressed as MWCNT count.

Table 2. Fiber count of MWCNTs in the cementitious matrix

	Fiber count
M	—
M + MWCNTs 0.1 wt %	36.1×10^{10}
M + MWCNTs 0.2 wt %	72.2×10^{10}

4.2. Testing procedure. Prismatic notched specimens $20 \times 20 \times 80$ mm were tested in three point bending at ages of 3, 7 and 28 days. The test was performed using a 25 kN MTS servo-hydraulic, closed-loop testing machine. The specimens were tested in a crack mouth opening displacement (CMOD)-controlled mode. The CMOD was used as the feedback signal to produce stable crack propagation at the rate of 0.008 mm/min, such that the peak load is reached in about 5 min. The prismatic specimens were monotonically loaded up to the maximum load. The applied load is then manually reduced after the load passed the maximum load and within the 95 % of the maximum load.

The two-parameter method [2] was used to obtain the fracture mechanics properties of the specimens from the experimental results. These properties include the critical stress intensity factor, K_{Ic}^S , the critical strain energy release rate, G_{Ic}^S , the critical crack

tip opening displacement, $CTOD_c$, and the critical crack length, a_c . For the application of the method the loading and unloading compliances and the peak load were monitored during the test. A typical loading-unloading load-CMOD curve of a mortar nanocomposite reinforced with well dispersed MWCNTs at an amount of 0.2 wt % of cement is shown in Fig. 1. According to the ASTM C348, specimens of strengths deviating by more than 10% of the average strength were discarded. The three-point bend specimen geometry and experimental setup is shown in Fig. 2.

5. Results and discussions. A series of experiments were performed to determine fracture mechanics parameters including critical stress intensity factor, K_{Ic}^S , critical strain energy release rate, G_{Ic}^S , critical crack tip opening displacement $CTOD_c$ and effective crack length a_c in notched three-point bend specimens. Specimens of neat mortar and mortar reinforced with MWCNTs at amounts of 0.10 and 0.20 wt % were performed. Fig. 3 presents the variation of K_{Ic} versus time for up to 28 days for neat mortar and mortar reinforced with MWCNTs at amounts of 0.1 and 0.2 wt % of cement.

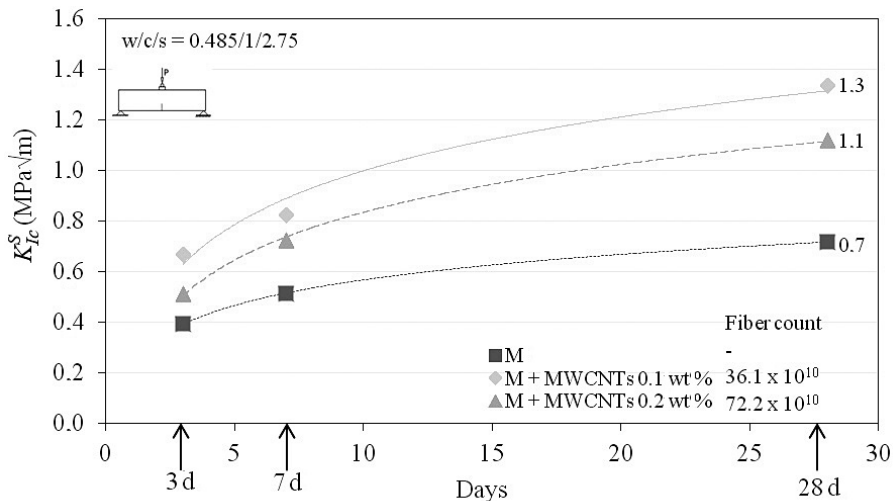


Fig. 3. Critical stress intensity factor, K_{Ic} , of neat mortar and mortar reinforced with MWCNTs at amounts of 0.1 and 0.2 wt % of cement versus time for up to 28 days.

The water to cement to sand ratio was $w/c/s = 0.485/1/2.75$. Note that K_{Ic} increases with time for all three cases. K_{Ic} also increases with the addition to neat mortar of MWCNTs. However, values of K_{Ic} are higher for an addition of 0.1 wt % than an addition of 0.2 wt % MWCNTs. This behavior can be attributed to poor dispersion of MWCNTs in mortar as their percentage increases. Analogous results for the critical strain energy release rate, G_{Ic} , are shown in Fig. 4.

Results for the critical crack tip opening displacement, $CTOD_c$, at 28 days for mortars reinforced with MWCNTs at amounts of 0.1 and 0.2 wt % are shown in Fig. 5.

Note that $CTOD_c$ at 0.1 wt % is higher than at 0.2 wt %. The bars in the figure indicate the error in the determination of $CTOD_c$. Finally, Fig. 6 presents results for the material length Q of 28 day neat mortar and mortars reinforced with 0.1 and 0.2 wt % of cement.

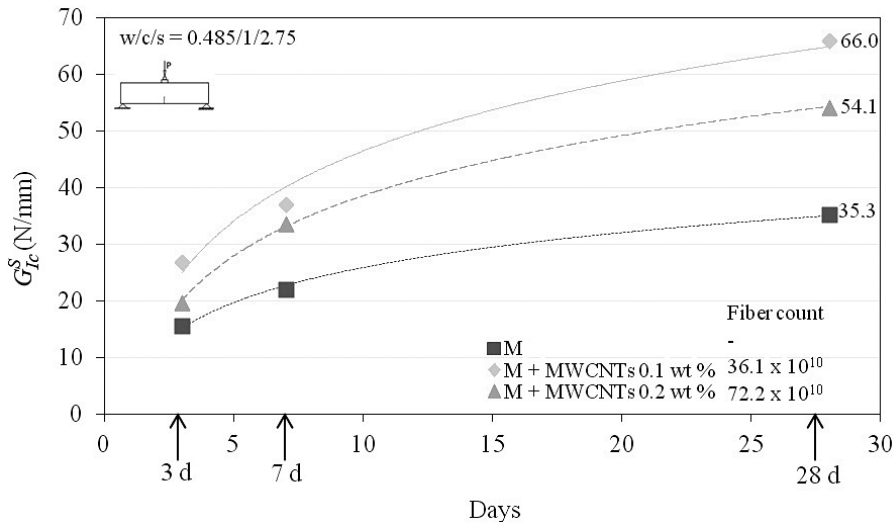


Fig. 4. Strain energy release rate of neat mortar and mortars reinforced with MWCNTs at amounts of 0.1 and 0.2 wt % of cement up to 28 days of hydration.

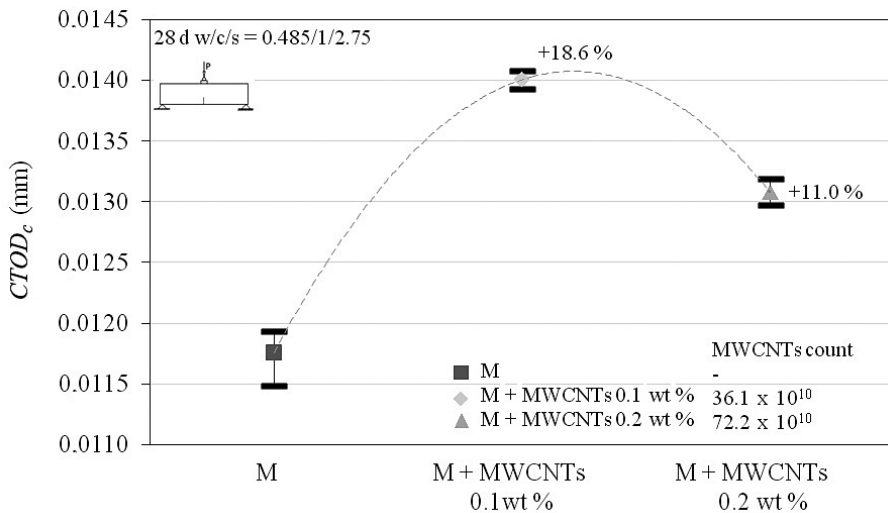


Fig. 5. Critical crack tip opening displacement of 28 days neat mortar and mortars reinforced with 0.1 and 0.2 wt % of cement MWCNTs.

6. Conclusions. A thorough study of the effect of reinforcement of neat mortar with multiwall carbon nanotubes (MWCNTs) at amounts of 0.1 and 0.2 wt % on the fracture behavior of the nanocomposite was conducted. Fracture properties measured include critical stress intensity factor, critical strain energy release rate, critical crack tip opening displacement and effective crack length. All these quantities were substantially increased by addition of MWCNTs and CNFs. Previous results [11–13] have demonstrated the beneficial effect of enhancement of tensile strength and Young's modulus of cement pastes, mortars and concrete reinforced with small amounts of MWCNTs and CNFs.

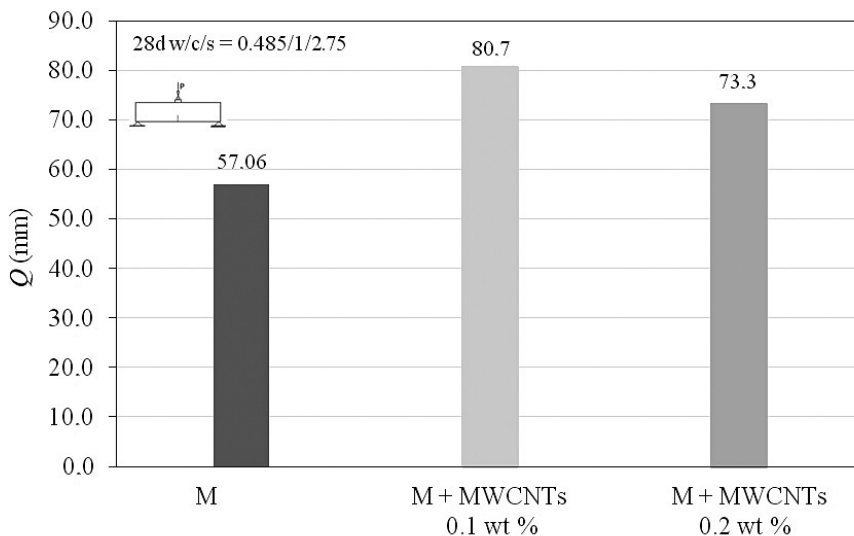


Fig. 6. Material length Q of 28 day neat mortar and mortars reinforced with 0.1 and 0.2 wt % of cement.

The main results of the present study referring to reinforcing capabilities of MWCNTs and CNFs at amounts of 0.1 and 0.2 wt % on the fracture properties of mortars may be summarized as:

- (i) the critical stress intensity factor increases by an amount of 128.6 %;
- (ii) the critical strain energy release rate increases by an amount of 154.9 %;
- (iii) the critical crack tip opening displacement increases by an amount of 39.7 %;
- (iv) the effective crack length increases by an amount of 10.3 %;
- (v) reinforcement of mortars by adding MWCNs at amount of 0.1 % is more effective than adding MWCNs at amount of 0.2 %. This behavior can be attributed to the poor dispersion of MWCNs at higher concentrations.

This paper is dedicated to Professor Nikita Fedorovich Morozov on the occasion of his 90th birthdate. We wish our friend Nikita many more blessed years with good health, happiness and productivity.

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Механика разрушения полученных по растворной технологии нанокомпозитов, армированных углеродными нанотрубками

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Целью данной статьи является исследование характеристик механики разрушения нанокompозитов, изготовленных по растворной технологии, армированных многостенными углеродными нанотрубками (MWCNTs). Коэффициент интенсивности критического напряжения K_{Ic} , скорость выделения энергии деформации GI_c , эффективная длина трещины a_c и смещение раскрытия вершины трещины $CTOD_c$ определяются с использованием образцов с трехточечным изгибом. Было установлено, что армирование раствора MWCNTs обеспечивает существенное улучшение вышеуказанных характеристик механики разрушения.

Ключевые слова: механика разрушения, многостенные углеродные нанотрубки, коэффициент интенсивности напряжений, скорость выделения энергии, смещение вершины трещины, нанокompозиты строительных растворов, цементирующие материалы.

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