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Portland cement mortar nanocomposites at low carbon nanotube and carbon nanofiber content: A fracture mechanics experimental study

Emmanuel E. Gdoutos, Maria S. Konsta-Gdoutos*, Panagiotis A. Danoglidis

Department of Civil Engineering, Democritus University of Thrace, Xanthi, Greece

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ABSTRACT

A thorough fracture mechanics characterization of Portland cement mortars reinforced with multi wall carbon nanotubes (MWCNTs) and carbon nanofibers (CNFs) took place. The critical values of stress intensity factor, K^S_{IC}; strain energy release rate, G^S_{IC}; crack tip opening displacement, $CTOD_C$; and critical crack length, a_c of 3, 7, and 28 days Portland cement mortars, reinforced with well dispersed carbon nanotubes and carbon nanofibers were experimentally determined. Prismatic notched specimens of neat mortars and mortars reinforced with 0.1 wt.% CNFs, and 0.1 and 0.2 wt.% MWCNTs were subjected to a three point closed loop bending test, using the crack mouth opening displacement, CMOD, as the feedback signal. The fracture parameters of the nanoreinforced mortars were then determined using the two parameter fracture model. The excellent reinforcing and toughening efficiency of MWCNTs and CNFs is demonstrated by a significant improvement in the critical stress intensity factor/fracture toughness (128.6%), critical strain energy release rate (154.9%), and critical crack tip opening displacement (39%). These results allow us to conclude that the MWCNTs and CNFs beneficially alter the nanostructure of the mortar matrix, resulting to a significant enhancement of all fracture and mechanical properties and provide the material, with the ability of performing multiple structural functions.

1. Introduction

The most important properties a structural material can provide are strength, stiffness, fracture toughness, energy absorption capability and ductility. Mortars and concrete are generally considered as inherently brittle materials; for that reason it is extremely difficult to improve simultaneously all four of the aforementioned structural properties. Multi wall carbon nanotubes (MWCNTs) and carbon nanofibers (CNFs), with their supreme stiffness, high strength and aspect ratio are excellent reinforcing candidate materials, offering unprecedented improvements in both strength and stiffness [1–3]. These fibers typically exhibit a Young's modulus around 1 TPa, tensile strength of 65–93 GPa, and maximum strain of 10–15% [4]. Reinforcement at this scale, in addition to providing fracture resistance was found to beneficially alter the nanostructure of cement based materials [5].

However, whilst most studies so far concentrate in experimentally determining the strength and the stiffness of nanoreinforced cement pastes, there is practically no information available in the literature on the determination of the fracture parameters such as fracture toughness, or strain energy release rate of MWCNTs or CNFs reinforced cement pastes, mortars or concrete. Fracture mechanics characterization of cementitious materials is of great importance for several 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 in the material. The energy required to create new material surfaces during the fracture process is a fundamental characteristic quantity of the material: the evaluation of the fracture mechanics parameters provides a basis for the understanding of the fracture process. Second, the design of large structures, like dams and nuclear reactors that behave in a rather brittle manner, as well as applications of high strength concrete with compressive strength higher than 100 MPa are benefited tremendously by fracture mechanics: classical theories of fracture 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. Moreover, the toughness as calculated by the area under the stress-strain curve of the material cannot be used as a true material property, since it is size and geometry dependent.

* Corresponding author.
E-mail address: mkonsta@civil.duth.gr (M.S. Konsta-Gdoutos).

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Direct application of fracture mechanics principles has been used for the experimental determination of the critical fracture toughness of cementitious materials. The foundations of the application of fracture mechanics to cementitious materials were laid down by the pioneering work of Hillerborg [6,7] who introduced the fictitious crack model of concrete, in an analogous way to the Dugdale-Barenblatt model of metals. The methodology has been similar to the experimental determination of the critical strain energy release rate, G_{IC}^{S}, or stress intensity factor, K_{IC}^{S}, in metals [8–10]. 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 mouth opening displacement (CMOD) response was recorded. The value of G_{IC}^{S}, or K_{IC}^{S}, is determined from the peak load or the load at the intersection with the secant of slope 95% of the initial slope and the initial notch length [11–15]. The values of G_{IC}^{S}, or K_{IC}^{S} obtained were dependent on the size of the specimen and its geometrical configuration [10,11,15–18]. However, since the experimental values of the critical strain energy release rate are specimen size dependent they could not be regarded as a characteristic material property. In both metal and concrete structures, nonlinear zones of small (small scale yielding approximation is treated by linear elastic fracture mechanics) or normal sizes (ductile fracture) develop at the crack tip [19]. In ductile metals however, the material in the nonlinear fracture process zone (FPZ) ahead of the crack tip undergoes hardening or perfect plasticity, whereas in concrete the material undergoes softening damage. In cementitious materials, within 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 FPZ ahead of the stress-free crack depends on the geometry and the size of the structure and the type of the material [20]. For cement paste the FPZ length is of the order of a millimeter, for mortar is about 30 mm, for normal concrete with medium aggregates 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 ozide ceramic is of the order of 0.1 mm, and in a silicon wafer of the order of 10–100 nm. Hence, direct application of linear elastic fracture mechanics for the characterization of the fracture behavior of cementitious materials may lead to erroneous results. One method developed to account for the FPZ in concrete materials is the Two-Parameter Fracture Method (TPFM) developed by Jenq and Shah [21]. The method takes into account that a change in specimen compliance can be correlated to the length of the effective crack. This effective crack length takes into consideration the inelastic phenomena that take place in the FPZ ahead of the crack tip at the critical (i.e., peak) load.

To the authors' knowledge only one reference [22] exists in the literature on the experimental determination of the fracture parameters of concrete or mortars reinforced with MWCNTs using the TPFM. Stynoski et al. studied the fracture properties of various Portland cement mortars containing silica fume, carbon nanotubes and carbon fibers using notched three-point bend specimens and the TPFM. They observed that carbon nanotubes provided a slight improvement in flexural strength and fracture toughness of about 5–10% at 7 and 28 days of age. Their effect on critical crack tip opening displacement ($CTOD_c$) was more significant, achieving a 20% improvement at 28 days. Using silica fume and carbon nanotubes together, a significant improvement in toughness and CTODc of about 35% and 56% after 28 days was observed.

Some information exists on the fracture properties of microfiber reinforced concrete. Taha et al. [23] used the effective crack model developed by Karihaloo and Nallathambi [24] to determine the critical energy release rate for high performance concrete, mortar, fiber reinforced concrete and masonry units. Other authors examined the fracture properties of mortars or concrete with fly ash, clay and metacaolin. Moukwa et al. [25] studied the effect of alumino-silicate clays on the critical stress intensity factor and $CTOD_c$. They concluded that appropriate use of silica fume and alumino-silicate clays can increase the ductility and strength of cementitious materials. Das et al. [26] 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. [27] 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. [28] studied the fracture characteristics of self-compacting concrete using notched three-point bend specimens. All the above results are summarized and compared with results of this study in Table 1.

It is the objective of the present work to provide a thorough fracture mechanics characterization through the experimental determination of the fracture parameters of nanomodified Portland cement mortars, reinforced with well-dispersed MWCNTs and CNFs and demonstrate their superb reinforcing and toughening effect. For this reason, three-point bending notched specimens of

Table 1
Comparison of the stress intensity factors of cement based materials.

Researchers	K_{IC}^{S} (MPa•√m)
Stynoski P. et al. [32] w/c/s = 0.485/1.0/2.75 25 × 25 × 125 mm 7 mm notch (a_o/W = 0.28) 28 d	0.41
Reda Taha M.M. et al. [33] normal strength mortar (MTR) 100 × 75 × 350 mm 25 mm notch (a_o/W = 0.25) 28 d	1.00
Moukwa M. et al. [35] w/c/s = 0.3/1.0/2.6 75 × 75 × 200 mm 25 mm notch (a_o/W = 0.3) 28 d	0.85
Das S. et al. [36] water/powder = 0.4 76 × 25 × 330 mm 19 mm notch (a_o/W = 0.25) 28 d	0.88
Sarker P.K. et al. [37] w/c/s = 0.47/1.0/2.61 100 × 100 × 600 mm 25 mm notch (a_o/W = 0.25) 28 d	0.52
Nikbin I.M. et al. [38] w/c/s = 0.48/1.0/5.0	1.12
Shah S.P. et al. [14] w/c/s = 0.45/1.0/2.6 76 × 28.6 × 305 mm 25 mm notch (a_o/W = 0.3) 90 d	0.89
Gdoutos E.E. et al. [Present work] w/c/s = 0.485/1.0/2.75 20 × 20 × 80 mm 6 mm notch (a_o/W = 0.3) 28 d	0.72