Construction and Building Materials 48 (2013) 265-269

Contents lists available at SciVerse ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Effect of the cementitious paste density on the performance efficiency of carbon nanofiber in concrete nanocomposite



PIALS

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HIGHLIGHTS

• Carbon nanofibers used in conjunction with steel or PVA fibers in high and ultra-high-performance concrete.

• Micro- and nano-scale reinforcement were found to render complementary effects in dense cementitious matrices.

• Optimum combination of CNF with steel fiber performs better than the optimum hybrid CNF/PVA systems.

ARTICLE INFO

Article history: Received 3 May 2013 Received in revised form 25 June 2013 Accepted 28 June 2013 Available online 28 July 2013

Keywords: Carbon nanofiber High strength concrete Ultra high performance concrete Steel fiber PVA fiber Mechanical properties

ABSTRACT

The large specific surface area, relatively active surface chemistry and close spacing of carbon nanofibers (CNFs) make them particularly effective in controlling the inception and growth of microcracks in cementitious materials. Given the fine geometry of nanofibers, their interactions with cementitious matrices would benefit from increased packing density and reduced size of pore system in matrix. These favorable features for effective interactions with CNFs are provided by high-strength (HSC) and especially ultrahigh-performance concrete (UHPC). An optimization experimental program was conducted in order to identify the desired combination of nano-/micro-scale reinforcement. The performance efficiencies of the desired nano-/micro-scale (hybrid) reinforcement in HSC and UHPC were evaluated and compared. The desired nano-/micro-scale reinforcement system comprising CNF and PVA fiber generally produced higher relative gains in the engineering properties of UHPC than HSC. Two different (PVA and steel) fibers were evaluated in conjunction with CNF in UHPC. The desired combination of steel fiber with CNF had higher fiber volume fraction than the desired combination of steel fiber with CNF. The desired nano-/micro-scale reinforcement with steel fiber performed better than that with PVA fiber in HSC.

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1. Introduction

Concrete materials may be categorized as normal-strength concrete (NSC) (with \leq 40 MPa compressive strength), high-strength concrete (HSC) (with 40–120 MPa compressive strength), and ultra-high performance concrete (UHPC) (with >120 MPa compressive strength) [1,2]. Concrete materials of higher strength exhibit a growing tendency towards brittle modes of failure. Introduction of fibers and more recently nanomaterials offer effective means of enhancing the toughness [3] and impact resistance [4] of HSC and UHPC materials. Nano-/micro-scale reinforcement systems comprising nano- and microscale fibers have been found to be particularly effective in concrete [5–7] The relatively high cementitious binder content and the high packing density of high-strength and especially ultra-high-performance concrete favor the dispersion and interfacial interactions of discrete reinforcement systems [8,9].

The discrete reinforcement system used in concrete are generally fibers of micrometer-scale (or larger) diameter [8,10]. At practical volume fractions (~1.0%), the spacing between those fibers is relatively large. Microcracks can initiate and grow freely in the space between conventional fibers before encountering them [11–14]. Nano-scale reinforcement can effectively fill the space between micro-scale fibers in order to effectively inhibit formation and early growth of cracks in concrete [15]. Examples of such nano-scale reinforcement include carbon nanotube (CNT) [16,17], carbon nanofiber (CNF) [13,18,19], and graphite nanoplatelet [20,21]. Uniform dispersion and adequate interfacial interaction are prerequisites to successful use of nano-scale reinforcement in cementitious and other nanocomposites [17,22–25]. Research into cementitious nanocomposites has largely focused on the use of cementitious paste without aggregate as matrix [11,19].

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^{0950-0618/\$ -} see front matter @ 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.conbuildmat.2013.06.094

Graphite nanomaterials provide large specific surface areas for interaction with cement hydrates. Their nano-scale cross-sectional dimensions (diameter/thickness) and micro-scale length, however, could compromise effective interactions with the porous structure of cementitious paste. The relatively high capillary porosity (typically more than 10 vol.%) of cementitious paste, a large fraction of which approach micro-scale dimensions, and also the relatively strong presence (~35 vol.%) of micro-scale crystals (calcium hydroxide, calcium aluminate hydrate, calcium monosulfate aluminate hydrate) which cannot effectively interact with nanomaterials, could hinder effective interactions of graphite nanomaterials with and thus their beneficial effects in conventional cementitious matrices. Only the (nanostructured) calcium silicate hydrate (C-S-H) offers the potential for development of massive (potentially primary) bonds with properly modified graphite nanomaterials. Fig. 1a schematically depicts a graphite nanomaterial occurring largelv outside C–S–H (with effective binding qualities), within capillary pores and in contact with micro-scale crystals; this nanomaterial cannot effectively interact with the cementitious paste. It may still render physical benefits (e.g., towards enhancement of the diffusion resistance and conductivity of cementitious matrix), but would not effectively contribute to the mechanical performance of the cementitious matrix. One can overcome this drawback by employing some practices which are commonly employed in higher-strength and ultra-high performance concrete mix design. The porosity and the volume fraction of micro-scale crystalline constituents of concrete can be reduced, and the capillary pore size distribution can be shifted towards nano-scale dimensions for effective interactions with nanomaterials (Fig. 1b) through: (i) maximizing the packing density [26] of the particulate matter in concrete while retaining the desired level of fresh mix workability; (ii) inducing pozzolanic reactions [27] which replace micro-scale crystalline products of cement hydration with C-S-H of reduced density to effectively fill capillary pores and bond to nanomaterials; and (iii) lowering the water/cementitious ratio by effective use of (normal or high-range) water reducers, which lower the size and content of capillary pores and also benefit dispersion of nanomaterials.

In the work reported herein, the reinforcement efficiencies of carbon nanofibers (CNFs) in HSC and UHPC are compared when CNFs are used in conjunction with micro-scale reinforcement. The engineering properties which are evaluated include flexural strength, energy sorption capacity, maximum deflection, impact resistance, abrasion resistance, and compressive strength. Optimization dosage of nano- and micro-scale reinforcement system in HSC and UHPC were identified. Two types of micro-scale fibers (steel and PVA) were considered in conjunction with CNF in UHPC.

2. Experimental program

2.1. Materials

Oxidized carbon nanofibers (OX-CNF) were used in this study. These nanofibers have an outer diameters ranging from 60 to 150 nm, and lengths ranging from 30 to 100 μ m. They provide 50–60 m²/g specific surface area (SSA), ~1.95 g/cm³ specific gravity, >95% purity, 240 GPa elastic modulus, and 30 GPa tensile strength. Poly Acrylic Acid (PAA, average Mw ~100,000, 35 wt.% in H₂O) was physisorbed onto nanofibers using the procedure described in the following section in order to improve the dispersion and interfacial interactions of nanofibers in cementitious past. PVA fibers were 13 mm long with diameter of 100 μ m and density of 1.3 g/cm³. Copper-coated steel fibers with 175 μ m diameter, 13 mm length and density of 7.85 g/cm³.

2.1.1. High-strength and ultra-high-performance concrete materials

Type I Portland cement, undensified silica fume with ~200 nm mean particle size, ~15 m²/g specific surface area and $\geq 105\%$ 7-day pozzolanic activity index, granite coarse aggregate with maximum particle size of 9.5 mm (see Table 1 for sieve analysis), natural sand (used in HSC) with maximum particle size of 2 mm (see Table 2 for sieve analysis), silica sand with maximum particle size of 0.5 mm (used in UHPC), and polycarboxylate-based superplasticizer (W.R. Grace, ADVA Cast 575) were the raw materials used for production of HSC and UHPC. The mix design for HSC and UHPC are presented in Tables 3 and 4, respectively. The targeted compressive strengths of HSC and UHPC were 70 and 150 MPa, respectively.

2.2. Carbon nanofiber preparation and dispersion in mixing water

Polyacrylic acid (PAA) with high density of COOH groups was considered for wrapping the CNFs in order to improve their dispersion in water and interaction with cement hydrates. For this purpose, CNFs were dispersed in water in the presence of polymer (PAA); a polymer-to-CNF weight ratio of 0.1:1.0 was considered [28]. CNFs were mixed with PAA 30% of the mixing water of concrete, and sonicated for 30 min. The resulting dispersion was exposed to microwave radiation for 10 min at 400 W, and stirred overnight.

Subsequently, the following procedure was implemented in order to disperse CNFs in concrete: (i) add the required amount of superplastisizer to the blend of oxidized nanofiber and PAA in water and stir for 12–15 h; (ii) sonicate the mix by repeating a cycle comprising (a) 10 min of sonication at 40%, 40%, 65% and 75% of maximal power (400 W) with 1-min breaks in between, (b) pulsing (1 min on, 30 s off) for 10 min at 80% of maximal power, and (c) repeating the previous (pulsing) step after 2 min of rest.

2.3. Sample preparation

Following the dispersion of CNFs in mixing water the ASTM D192 and C305, procedures were followed for preparation of the HSC and UHPC materials. These procedures involved: (i) mixing dry ingredients (cement, silica fume, sand, coarse aggregate) for 5 min in a mortar mixer (Hobart Model A200F) at speed 1; (ii) adding water (with dispersed nanomaterials) for 1 min at speed 1, 2 min at speed 2, and (while adding steel or PVA fibers) 2 min at speed 3; and (iii) casting concrete into molds per ASTM C192, and consolidating them on a vibration table (FMC Syntron Power Plus) at intensity 10. The specimens were moist-cured inside molds (ASTM C192) at room temperature for 20 h after casting, and then demolded and subjected to 48 h of steam curing at 70 °C. The specimens were then conditioned at 50% relative humidity and ambient temperature for 7 days prior to testing



Fig. 1. Schematic depiction of the conventional and refined microstructures of cementitious matrix incorporating a graphite nanomateiral (C = capillary pores; H = microscale crystalline products; A = calcium silicate hydrate).

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