Construction and Building Materials 86 (2015) 85–94

Contents lists available at ScienceDirect

Construction and Building Materials

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

A novel method for the determination of polymeric micro-fiber distribution of cementitious composites exhibiting multiple cracking behavior under tensile loading

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HIGHLIGHTS

- A methodology is proposed to determine cross-sectional micro-fiber distribution.
- Fiber distribution coefficients and fiber density maps were determined.
- The validity of method is checked by comparing two distinctive HTPP-ECCs.
- Relatively low matrix viscosity is beneficial for homogeneous fiber distribution.

ARTICLE INFO

Article history:
Received 13 December 2014
Received in revised form 13 March 2015
Accepted 23 March 2015
Available online 9 April 2015

Keywords:
Cementitious composite
Polymer fiber
Tensile ductility
Scanning electron microscopy
Multiple cracking
Fiber distribution

ABSTRACT

A methodology is proposed to measure the micro-fiber distribution of high tenacity polypropylene fiber reinforced engineered cementitious composites (HTPP-ECC). For this purpose, scanning electron microscope (SEM) images of failure section were captured at backscattered electron mode (BEC) and analyzed via a consecutive set of image processing algorithms. Fiber distribution coefficients and fiber density maps of each section were determined. The validity of fiber distribution parameters is checked by comparing the multiple cracking potential of two distinctive HTPP-ECCs from the view point of tensile ductility (M24: 3.1%, M37: 0.8%) and matrix rheology (modified Marsh cone flow time; M24: 23 s, M37: 40 s). Results showed that a better fiber distribution at failure section and relatively porous matrix structure improved the multiple cracking potential of composites. The proposed methodology is capable of detecting the fiber distribution variation at micro-scale. Relatively high matrix viscosity was found responsible for the undesirable polymeric micro-fiber distribution of HTPP-ECCs.

1. Introduction

Strain hardening cementitious composites (SHCCs) exhibiting multiple cracking behavior under tensile loading have been extensively studied to address the brittleness, ductility and energy absorption shortcomings of cementitious composites [1–3]. As a family of SHCCs, engineered cementitious composites (ECC) have been developed by Li and coworkers [4]. Depending on the fiber type, dosage, matrix fracture toughness, matrix flaw structure and fiber-matrix interaction, ECC with tensile ductility as much as 300 times higher than normal concrete can be designed for different purposes and functions [2,5]. These added functions to high ductility can be listed as lightweight [6], self-sensing [7], self-healing [8] and high strength [9] properties, respectively. A micromechanics based design tool set was developed for ECC design [2,4,10]. Details of the micromechanical model can be found in Li et al. [11].

First version of ECC was developed by using surface modified high-modulus polyethylene (PE) fibers (PE-ECC) with 1.5% fiber content [12]. However, broad application of PE-ECC was hindered by its relative high cost. Due to this fact, the applicability of high strength polyvinyl alcohol (PVA) fibers was extensively studied as a more economical alternative [13]. The cost of PVA fiber is about 1/8 that of high-modulus PE fiber [11]. With certain adjustments in fiber and surface properties, PVA-ECC that exhibits robust multiple cracking behavior was successfully developed even at moderate fiber dosages (2% by total volume) [14]. Previous studies on low strength and low cost polypropylene fibers showed that critical dosage for multiple cracking is at least equal or higher than 2%. This fiber dosage usually causes fiber dispersion difficulties at

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http://dx.doi.org/10.1016/j.conbuildmat.2015.03.094
0950-0618/

the stages of mixing and molding [15]. Recent progress in polymer spinning technology and experimental research efforts to modify the physical and mechanical properties of polypropylene fibers, which are known as high tenacity polypropylene (HTPP) fibers, brought a promising cost effective alternative to PVA and PE fibers, [16–18]. The cost of HTPP fibers is usually less than PVA fibers due to the world wide availability of PP raw material [17]. HTPP fibers have been employed in corrugated and flat cement based sheets formed with Hatschek process at relatively high dosages [19]. In recent years, applicability of HTPP fibers in cost effective ECC design have also been studied at moderate dosages (2% by total volume) [20,21].

Homogeneous and isotropic distribution of fibers in cementitious matrix is a crucial step for the preparation of ECCs will exhibit consistent and robust multiple cracking behavior under tensile loading. However, fiber dispersion efficiency of cementitious matrix mainly depends on the fresh rheology which influences both the feasibility and density of multiple cracking [22]. The relation between fiber bridging stress and crack width and in particular fiber bridging strength strongly depend on the fiber distribution characteristics [23]. For a specimen subjected to uniaxial tension, poor fiber distribution will lead to a reduced effective fiber dosage at the "weakest" cross-section within the specimen. An inappropriate effective fiber dosage will lead to a lower fiber bridging strength at the weakest section, thus fiber bridging capacity exhausts earlier [22]. If fiber distribution at the "weakest" cross-section within the specimen is known, it is possible to check and evaluate the multiple cracking potential of composites.

Fiber distribution characterization techniques are mainly focused on metallic fibers, since steel fibers are mainly utilized in fiber reinforced composites. High resolution digital images captured from cut and polished cross-sectional areas of specimens are used for this purpose. Since the diameters of steel fibers are usually higher than 0.15 mm, it is possible to detect fibers from images with appropriate resolution by an optical microscope equipped with a simple CCD camera [24,25]. Light reflecting properties of steel fibers also promote the contrast between fiber and surrounding matrix as an advantage over polymeric fibers [25]. Finally the distribution of fibers and their orientation can be determined by suitable set of image processing commands [3]. Alternatively, non-destructive monitoring methods of fiber distribution and orientation, that are based on X-ray transmission photography [26,27], X-ray computed tomography (CT) [28,29] and AC-impedance spectroscopy (AC-IS) [30] have been previously used in order to correlate the rheology and mechanical properties of composites incorporating conductive fibers. However, the methods listed above are usually not suitable for polymeric fibers especially with diameter less than 50 μm. The low contrast between polymeric fibers and cementitious matrix makes it difficult to distinguish fibers with conventional optical microscope techniques. This problem was overcome by using fluorescence imaging techniques in the case of PVA fibers which are usually employed in ECC compositions [22,23,31]. PVA fibers irradiated with a UV filtered light source (mercury discharge lamp emitting light over a broad spectrum (UV, Visible, and Infrared)) of predetermined wavelength fluoresce and can be distinguished from the dark surrounding cementitious matrix with a low background signal [23]. However, this method is not applicable for HTPP fibers to emit visible light (wavelength > 400 nm) since the excitation and emission bands of PP (285 and 305 nm, respectively) are much lower than PVA fibers (380 and 447 nm). The optimum excitation and emission wavelength bands of PVA, PET, PE and PP polymers, which are proposed on the basis of relative fluorescence intensity, can be found in Lee et al. [32].

Due to the problems faced with conventional and advanced light microscope techniques mentioned above, an alternative imaging methodology, based on scanning electron microscopy (SEM), is planned to use in order to visualize HTPP fibers. SEM has been extensively used to monitor the hydration process in cement based composites. It is possible to distinguish various zones by contrast difference between the hydrated (darker) and anhydrous (lighter) phases [33]. However, this method was rarely used to differentiate polymeric fibers from the surrounding matrix at a polished section. Takashima et al. [34] used SEM to examine the fiber orientation and fiber volume fraction of conventional polypropylene fiber reinforced composites manufactured by hot steam extrusion molding. Since fiber volume fraction of these composites were as high as 8.4% by total volume, extrusion molding oriented most of the fibers along the extrusion direction which improved the composite tensile performance at that direction. However, details of the SEM settings, specimen preparation, imaging and analysis methods are not explained in that paper. Akkaya et al. [35,36] applied some image analysis on SEM images to study the effect of dispersion and orientation of PVA fibers on the multiple cracking behavior and toughness of fiber-reinforced composites.

This study aims to quantify the HTPP micro-fiber distribution characteristics in cementitious composites by SEM analysis. An experimental program was conducted to evaluate variations in fiber distribution of two distinct HTPP-ECCs over the cross-section near the failure crack. For this purpose, cross-sectional analysis of specimens' failure sections were performed by capturing SEM images under backscattered electron (BEC) mode. The variation in fiber distribution was determined through a set of image processing commands applied on BEC–SEM images. The proposed methodology was used to quantify the fiber distribution coefficient of HTPP fibers for the first time. Furthermore, the role of fiber distribution at failure section on multiple cracking potential and tensile ductility of HTPP-ECCs was evaluated under tensile loading.

2. Experimental program

2.1. Mix proportions, materials and specimen preparation

Two distinctive matrices in terms of fiber dispersion ability were designed to prepare HTPP-ECCs incorporating the same amount of HTPP fibers (2% by volume). Mixture proportions are listed in Table 1. First matrix (coded as M24) was composed of Type I ordinary Portland cement (OPC) and class F fly ash conforming the requirements of ASTM C150 [37] and ASTM C 618 [38] standards, respectively. The chemical compositions of OPC and class F fly ash can be found in Felekoglu et al. [21]. Scanning electron microscope (SEM) observations showed that fly ash particles usually have spherical shape with smooth surface texture. Second matrix (coded as M37) was composed of the same type of cement in addition to a fine limestone powder (FLP) with calcium and magnesium carbonate contents of 96.5% and 1.6%, respectively. The average particle size of FLP was 10 μm and 97% of the powder was finer than 40 μm. Microscopic observations on FLP showed that this powder has rough and angular surface texture. A polycarboxylate-based high range water reducing admixture (HRWRA) was used in both matrices to achieve flowability. HTPP fiber with 12 μm diameter and 10 mm length was used in all mixtures. The density, nominal tensile strength, Young's modulus, and elongation percentage at rupture of HTPP fibers were 0.91 g/cm^3, 850 MPa, 6 GPa, and 21%, respectively.

HTPP-ECC mixtures were prepared using a Hobart mixer with 5 L capacity. All solid ingredients were premixed without water for 2 min. Water and HRWRA were then added. The resulting mixture was mixed for 1 min at low speed and for 2 min at high speed, respectively. The matrix flowability was indirectly measured using a modified Marsh cone (orifice diameter was increased to 20 mm) as described in Li and Li [22]. Modified Marsh cone flow time can be correlated by the plastic viscosity of matrix. A longer flow time indicates a higher plastic viscosity. Depending on the W/C ratio and HRWRA dosage the flow times of M24 and M37 were twofolds different. In the case of M24 mixture, a high W/C ratio is targeted (0.88) in order to balance the water requirement of high volumes of fly ash content. The HRWRA requirement of this matrix was only 7.4 kg/m^3 thanks to this high W/C ratio and spherical shape of fly ash particles which improve the workability of mixture (flow time: 23 s). However, a relatively longer flow time (40 s) is obtained in the case of M37 with a relatively lower W/C ratio despite the addition of high amounts of HRWRA (as high as 36.3 kg/m^3). The longer flow time indicates a relatively higher viscosity for M37 mixture. The source of high viscosity, even in the presence of high amounts of HRWRA, can be attributed to the rough and angular surface texture of