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Functionally-graded fiber-reinforced cement composite: Processing, microstructure, and properties

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Abstract

A functionally-graded material system has been developed with a spatially tailored fiber distribution to produce a four-layered, functionally-graded fiber-reinforced cement composite (FGFRCC). Fiber volume fractions were increased linearly from 0% in the compression zone to 2% in the tensile zone so that more fibers were available to carry tensile stress in a bending beam experiment. Extrusion was used to produce single homogeneous layers of constant fiber volume fraction and highly oriented fibers. The FRCC layers with different fiber volume fractions were stacked according to the desired configuration and then pressed to produce an integrated FGFRCC. Polished cross-sections of the FGFRCC were examined using a scanning electron microscope (SEM) to measure the fiber distribution. Flexural tests were carried out to characterize the mechanical behavior and to evaluate the effectiveness of the designed fiber distribution. No delamination between layers was observed in the fractured specimens. Compared to homogeneous FRCC with the same overall fiber volume fraction, the FGFRCC exhibited about 50% higher strength and comparable work of fracture.

Keywords: Fiber-reinforced cement composite (FRCC); Functionally-graded fiber-reinforced cement composite (FGFRCC); Extrusion; Functionally-graded material (FGM)

1. Introduction

Various kinds of fiber-reinforced cement composites (FRCC) have been developed, studied, and applied in construction in the past few decades. A few proceedings and books provide comprehensive information about FRCC [1–6]. Many kinds of micro-fibers have been commercially developed and are now being used in FRCC. A properly designed FRCC may offer enhanced mechanical behavior e.g. higher strength and higher toughness [2]. Generally, dense matrix and strong fiber–matrix bond are expected, so that not only high toughness, but also high strength can be achieved. Extrusion has been an effective way to produce FRCC [7–11]. Essentially, extrusion involves forcing a plastic material through a small die. When this technique is applied to manufacturing of cementitious materials, very low water to cement ratio must be utilized, which contributes to high strength of the matrix. A major advantage of using extrusion for FRCC is that the high shearing action developed during the forming process forces short fibers to be aligned in the extrusion direction. Such fiber alignment improves the mechanical performance of FRCC in the extrusion direction, which is beneficial for components that may carry tensile load only in one direction. Fiber alignment by extrusion has been studied quantitatively [9,12]. A recent study has shown that extrusion, compared with casting, can further enhance fiber–matrix interfacial bonding [13]. However, improvement in bonding may not be beneficial for the toughness of a FRCC [9,13,14], as demonstrated by studies in which interfacial bonding was tailored either by modifying fiber surface chemistry [14] or by modifying binder composition [13].

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One important issue, associated with the practical use of FRCC, is often the material cost, dominated by the cost of fibers [15]. Thus there is a need for new technology to make more effective and efficient use of costly fibers. The FRCC is homogeneous in a bulk scale, i.e. the fiber volume fraction is uniform, apart from potential unintended variations caused by the production processes (settlement, segregation, etc.). For certain FRCC components, a significant fraction of the fibers is not utilized to its full potential. One typical example is FRCC beams or panels under bending. In ordinary service situations without considering substantial cracking, near half of the volume of the beam is under compression, where the fibers usually do not contribute much to strength, and the compressive strength of the cementitious matrix itself is normally high enough to carry the load. In the tensile region, as tensile stress varies from zero at neutral axis to maximum stress through beam depth, fibers near neutral axis carry smaller portion of the tensile loading than those fibers that are away from the neutral axis. In this sense, many of the fibers are not used efficiently. In such a case, an efficient use of the fibers would be achieved if they were distributed according to the amount of load to be carried and if they were placed where they are needed the most. One example to use fibers more efficiently is to build sandwich panels from thin fiber-reinforced faces and a lightweight core [3, p. 143–51]. This is also similar to the case of repairing damaged beams or strengthening in-service beams, where carbon fiber sheets are usually bonded to the bottom of the beam. While this procedure can be effective, significantly different properties between the fiber sheets and the host concrete may cause potential delamination problems. This deficiency can be addressed by utilizing the concept of functionally-graded materials (FGM) [16] – i.e. by distributing a fixed amount of fibers according to proportion of load to carry, the graded composite may provide better performance than the homogeneous composite. Recently, preliminary investigations of functionally-graded fiber-reinforced cement or concrete composite (FGFRCC) were presented at the multiscale and functionally-graded materials (M&FGM 2006) conference and the reader is referred to the proceedings of the event [17–20].

The focus of the present work is on the processing procedure, microstructural verification and preliminary mechanical testing. A ram extruder was fabricated to manufacture thin FRCC layers with different fiber volume fractions. Then, extruded beams with increasing fiber volume fraction were stacked and pressed in a mold to integrate them into an FGFRCC. Stacking procedure is chosen because it is a straightforward way to generate functional gradation before subsequent processing is taken to integrate the stacked layers to form FGMs [21,22]. The critical issue of the fabrication process, the matching of paste plasticity properties between different layers, will be discussed later. For comparison, beams with a single global fiber volume fraction were also stacked and pressed. The cross-section of the FGFRCC was observed in a scanning electron microscope (SEM) using backscattered electron image (BEI). Through image processing of the SEM image, fibers were segmented from the matrix and voids to be examined. Three point bending tests were carried out to observe the bending behavior for both graded and homogeneous specimens. Interface conditions were examined in the fractured specimens. The bending response was compared between graded and homogeneous beams.

2. Materials

The main components of the FGFRCC were Type I ordinary portland cement, fly ash, polyvinylalcohol (PVA) fiber, superplasticizer, rheological modifier and water. Except for the portland cement, the materials were selected to facilitate the fabrication technique, a combination of extrusion and pressing.

Fly ash is widely used as pozzolanic material in making concrete or various kinds of cement-based materials. FRCC with high content fly ash made by extrusion has been studied [23]. The main reason for adding fly ash in the mix was to improve the plastic properties of the fresh paste so that it is more extrudable, and also to reduce the strength of the bond between paste and fiber. The spherical morphology of fly ash particles is particularly beneficial to workability [24]. Also, when substantial amount of fly ash replaces cement, the overall heat of hydration and rate of heat generation reduces, which prolongs the plastic properties required for pressing. The fly ash used in this study is class F type.

The PVA fibers used in this study are from Kuraray Co., Ltd., Japan.[1] Its type is REC7, with an available cut length of 6 mm and a diameter of 27 μm. The properties of PVA fibers are shown in Table 1. We used PVA fibers because their special surface chemistry and the fibers produce a workable mixture. PVA fibers have been successfully used in manufacturing of FRCC [13]. The surface chemistry of PVA fibers is hydrophilic so that a strong chemical bond can be developed with the hardened cementitious matrix [25]. The PVA fibers are soft so that they will not clog the die entrance, and pliant so that they will not be broken during extrusion.

Superplasticizer was added to reduce the ratio of water to cementitious materials, and to enhance the fluidity of the fresh paste. The superplasticizer used in this study is Grace ADVA® 100 superplasticizer. It is an aqueous solution and has a solid content of 35% by mass.

Rheological modifier was added to improve cohesion in the fresh paste to make it dough-like and also to prevent separation of water during extrusion, so the FRCC extrudate can retain its shape under its own weight. The rheological modifier used in this study was METHOCEL F4M, a hydroxypropyl methylcellulose (HPMC) manufactured by the Dow Chemical Company, USA. Its liquid

[1] http://www.kuraray.co.jp/en/business/fibers.html.

Table 1
Properties of PVA fiber (provided by Kuraray Co., Ltd.)

Cut length (mm)	Diameter (μm)	Density (g/cm^3)	Tensile strength (MPa)	Elongation (%)	Tensile modulus (GPa)
6	27	1.3	1600	6	37

solution shows shear thinning properties that facilitates the paste flow in the extruder during extrusion [10]. The use of cellulose has been popular in formulating cementitious mixtures for enhancing extrudability, whether or not they contain fibers.

3. Experiments

3.1. Mix proportions

For the FGFRCC, the fiber volume fractions for the four layers were: 0%, 0.67%, 1.33% and 2%, respectively. Therefore, the total fiber volume fraction of the FGFRCC is 1%. The basic mix proportions of different layers are shown in Table 2. The specific 4 mix proportions for FGFRCC were chosen based on many trial mixes and extrusions. The primary goal was to minimize the paste plasticity difference between layers so that pressing would produce uniform FGFRCC beam components. How to select mix proportions for FGFRCC fabrication is discussed in a later section.

3.2. Mixing procedure

A rotary planetary-type mortar mixer was used for mixing. The dry ingredients were first mixed for at least 1 min. Water was then added slowly, after which mixing was continued for 5–10 min to reach a dough-like state with no obvious fiber bundles. The mixing time needed to reach this state was found to depend on dosage of HPMC, fiber volume fraction and water to binder ratio. Usually a dosage of HPMC was selected such that all formulations could be mixed to a dough-like state in 5–10 min irrespective of fiber fraction.

3.3. Extrusion

The fabricated extruder is shown schematically in Fig. 1. The extruder was designed to be assembled and disassembled quickly so that each extrusion could be finished promptly. The ram speed was fixed at 20 mm/min for an extrudate speed of 255 mm/min.

Table 2
Mix proportions of FGFRCC layers

Cement (wt.)	Fly ash class F (wt.)	Water (wt.)	Fiber (vol.%)	Superplasticizer[a] (wt.)	HPMC/water (wt.)
0.7	0.3	0.23	0	0.002	0.04
0.7	0.3	0.23	0.67	0.002	0.04
0.7	0.3	0.23	1.33	0.002	0.04
0.7	0.3	0.23	2	0.002	0.04

[a] Effective weight of superplasticizer (Grace advance flow).

The extrudate was cut to desired lengths (a cut length of 120 mm was used in this study) for subsequent pressing. Immediately after each extruded layer was cut, it was covered with a wet cloth to prevent drying, which would cause hardening of the extrudate surface and make subsequent pressing difficult.

3.4. Stacking and pressing

After all four layers were extruded, they were stacked as shown in Fig. 2, with the fiber volume fraction increased from 0% at the top to 2% at the bottom. The total thickness before pressing was 16 mm. The same procedure was used to make homogeneous FRCC with uniform fiber volume fraction by stacking four layers with the same fiber volume fraction to make one homogeneous FRCC beam. To reduce friction, the stacked layers were put in a rigid steel mold lined with smooth plastic sheet (Fig. 3).

The length of the stacked layers was three fourths the length of the mold which is 160 mm, so that pressing caused the FGFRCC to extend in length and correspondingly reduce in thickness. Through this deformation process, the stacked layers bonded to each other and formed a continuous beam. A strong inter-layer bond developed later through cement hydration, which is essential to obtain integral FGMs. Greater thickness reduction made it more likely that layers would be wavy. We found that 25% thickness reduction applied was sufficient to produce interlayer bonding strong enough to avoid delamination while producing layers of uniform thickness.

Pressing was carried out on an Instron 4500 electromechanical testing machine using displacement control. Displacement rate of 5 mm/min was used, and pressing of the FGFRCC was finished in about 1 min. The pressing force increased sharply when the pressed layers filled the volume of the mold. Pressing was stopped when the force caused an average pressure of about 1.80 MPa.

After each pressing operation, the FGFRCC with its plastic sheet was removed for curing. The plastic sheet mold served to prevent drying and gave support to the soft FGFRCC.

3.5. Curing

After extrusion and pressing, all extrudates with plastic mold were covered by a plastic sheet for 1 day. Then the plastic molds were removed and the specimens were fast cured at 90 °C in a water bath for another 2 days. After this high temperature curing, the specimens were kept at ordinary laboratory conditions for 48 h. Before flexural testing, the specimens were oven dried at 105 °C for 1 day and kept at laboratory condition for another 24 h. The timing of the flexural testing was 7 days after the specimens were fabricated. The curing scheme is the same as that used by Shah

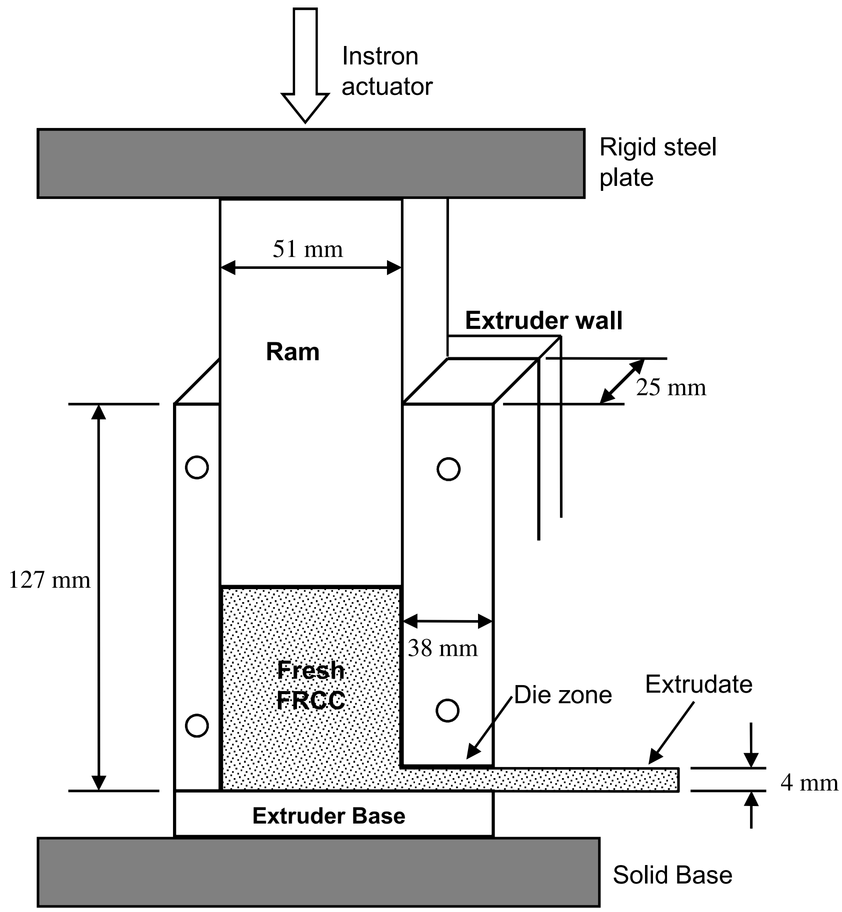

Fig. 1. Schematic illustration of a cross-section of the actual extruder developed in the present research.

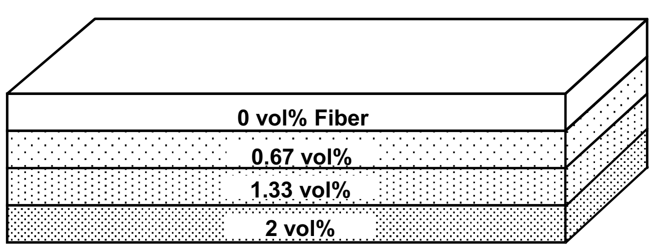

Fig. 2. Stacked extruded layers with varying fiber volume fraction.

and colleagues [12,13]. It was adopted in this study to reduce the time to subsequent testing.

3.6. Scanning electron microscopy observation

Cross-sectional surfaces of FGFRCC were directly cut and polished to observe the fiber distribution and the interface between layers. The surfaces were examined using SEM backscattered electrons image (SEM/BEI). In order to cover an observation field containing all layers, several consecutive images were taken in the gradation direction. The images were then combined into a panorama and analyzed using an image processing software, ImageJ,[2] to show fiber gradation.

[2] http://rsb.info.nih.gov/ij/.

3.7. Flexural testing

Three-point bending tests were carried out using an Instron testing machine to characterize the deflection behavior of the homogeneous and graded specimens. The FGFRCC and homogeneous FRCC beams have a nominal dimension of: width 25 mm, depth 12 mm and length 160 mm. The span was set to 120 mm so that no further cutting was needed. Displacement was controlled at a cross-head speed of 0.3 mm/min.

4. Mechanical properties

4.1. FRCC properties

The computed equivalent elastic flexural stress is plotted versus stroke displacement in Fig. 4. The first cracking stress, f_{fc}, the maximum flexural stress, f_{max}, the corresponding displacement, d_{max}, and the work of fracture measured from this figure are summarized in Table 3. The first cracking stress, f_{fc}, is defined as the stress at the end of the stress–displacement linearity, sometimes called bend-over-point (BOP). The estimated work of fracture is defined here as the area under the flexural stress versus displacement curve. Each value is an average test result of

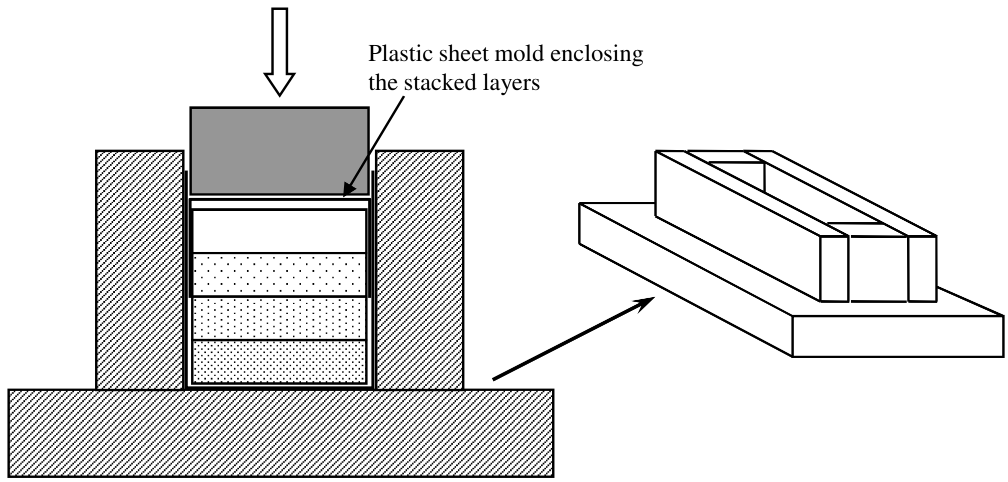

Fig. 3. Mold for pressing the stacked layers to produce FGFRCC.

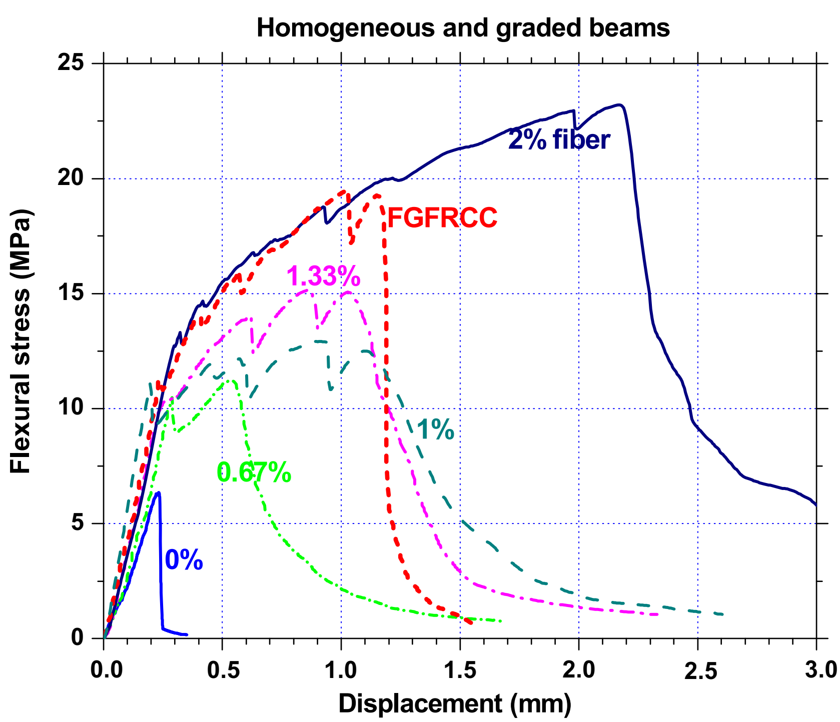

Fig. 4. Flexural response of homogenous beams with varying fiber volume fractions, and of FGFRCC with 1% overall net fiber volume fraction.

Table 3
Summary of bending tests of homogeneous FRCC and FGFRCC

	0%	0.67%	1%	1.33%	2%	FGFRCC
f_{fc} (MPa)	5.72	11.46	10.56	9.870	12.12	11.14
f_{max} (MPa)	5.72	11.83	12.73	14.65	22.94	18.45
d_{max} (mm)	0.18	0.545	1.01	0.97	2.06	1.19
Work of fracture (MPa mm)	0.573	5.719	16.64	15.71	46.15	17.45

three specimens. All FRCCs show deflection hardening response [26]. The effect of fiber volume ratio on strength and the work of fracture are self-evident from Fig. 4, both increased with fiber volume ratio.

As shown in Fig. 5, with addition of fibers, the FRCC and FGFRCC beams have a f_{fc}, or BOP, about two times of the f_{fc} of the beam without fibers. Moreover, it is observed that all FRCCs and FGFRCC have a similar

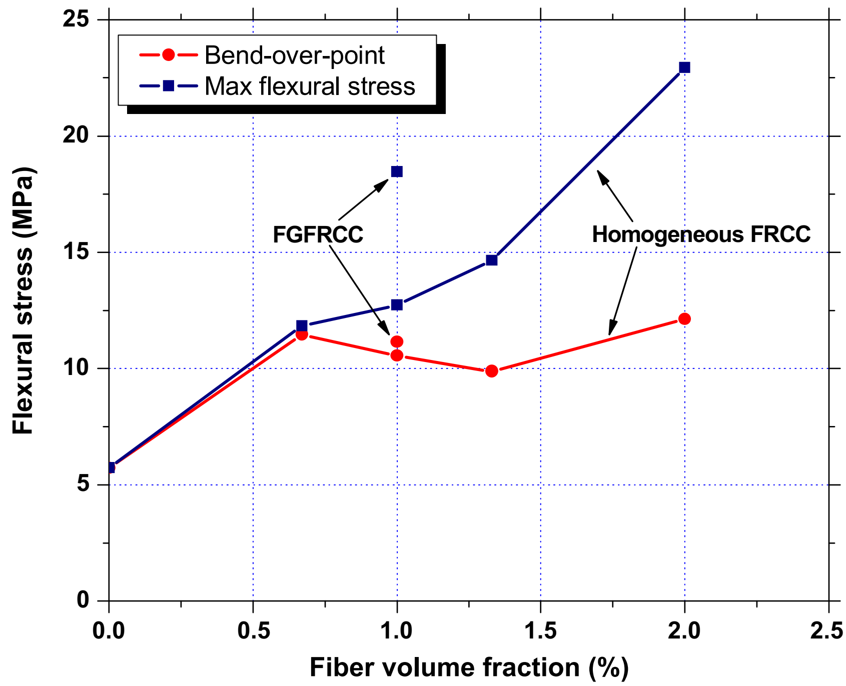

Fig. 5. First cracking stress and maximum flexural stress of homogeneous FRCC and FGFRCC.

value of f_{fc}, which is about 10–12 MPa. The stress for FRCC with 2% fiber increased much more after the BOP, compared to FRCC with small fiber volume fraction.

4.2. FGFRCC properties

Figs. 4–6 also show the flexural response of the FGFRCC. Compared to homogeneous FRCC, after the BOP, the increase of flexural stress with displacement most closely resembles that of 2% FRCC, up to the mid-point of the hardening part of the 2% FRCC. This can be expected because the response of the FGFRCC is predominantly controlled by its bottom layer, which contained 2% fibers. However, the descending part of the curve dropped more sharply in the FGFRCC because of the relatively low fracture toughness of the upper layers. The softening curves

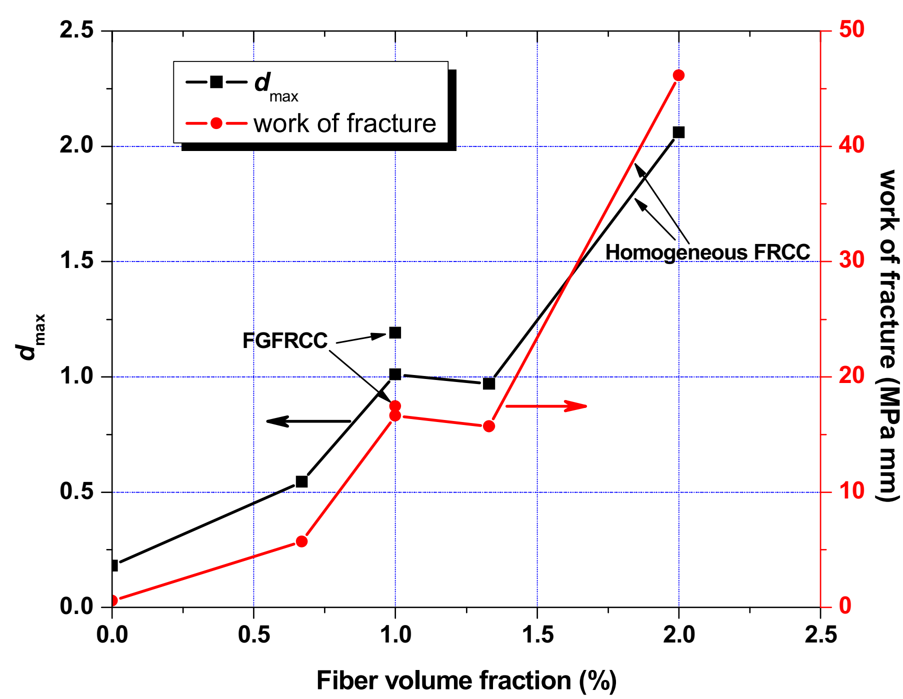

Fig. 6. Comparison between homogeneous FRCC and FGFRCC.

observed in the homogeneous FRCC were not observed for the FGFRCC. This demonstrates that failure of the middle two layers was no later than the bottom layer.

The fact that FGFRCC demonstrates deflection hardening behavior can be appreciated by comparing it to a recent investigation by Shin et al. [27]. In their experiments, they tested the flexural behavior of a binary system of plain concrete and ductile fiber-reinforced cementitious composite (DFRCC) with the DFRCC on the tension side. Their results show that the relative lack of toughness of the plain concrete above the DFRCC layer leads to the abrupt loss of beam strength when the DFRCC layer fails, which is similar to what is seen of the FGFRCC in Fig. 4. However, their material system does not show hardening behavior beyond BOP. Moreover, the brittle failure of the plain concrete is prior to the failure of the DFRCC layer. This contrast, apparently, indicates that the FGM solution is able to address such drawbacks typically seen in the binary systems.

The strength and work of fracture were greater for the homogeneous 2% FRCC than for the FGFRCC. This is ascribed to the fact that the former has four 2% FRCC "layers" while the latter has only one 2% FRCC layer at the bottom. However, the FGFRCC has an overall fiber volume fraction of 1%. Compared with homogeneous FRCC, FGFRCC with 1% total fiber volume fraction has higher strength than and comparable work of fracture values as those of 1.33% FRCC. The f_{max} of FGFRCC is about 50% and 30% higher than that of 1% and 1.33% FRCC, respectively. With 25% less fiber, the FGFRCC beam was able to sustain higher loads than the 1.33% FRCC beam before failure. A simple gradation allows the fibers to be used more efficiently. This result demonstrates that the idea of FGM for efficient use of fiber in FRCC is promising.

4.3. Reverse bending of FGFRCC

For the fabricated FGFRCC, the usual loading condition is that the 2% fiber layer is under tension. Tests were also performed to investigate the reverse situation when the 2% fiber layer is under compression. Fig. 7 shows a typical result. For comparison, the results of the normally loaded FGFRCC and the 1% homogeneous FRCC are also shown. As expected, the reversely loaded FGFRCC had a much lower first cracking stress. Only one crack is indicated by the single sharp drop in stress after the BOP. Its post-cracking maximum flexural stress is even lower than the first cracking stress, though for the second peak, the neutral axis moved up so that some of the fibers began to carry tensile load. In the normally loaded FGFRCC, the reduction of flexural stiffness is not as drastic due to the bridging effect from the fibers. Though it had low load carrying capacity, the reversely loaded specimen showed a long softening tail. The plateau of the tail developed as a result of bending of the ductile upper layers that have more fibers than the lower layers.

5. Microstructure

Fig. 8 shows a fracture surface of a hardened FRCC. The fracture surface is normal to the extrusion direction. The image clearly shows the high degree of fiber alignment along the extrusion direction. Fig. 9a shows the SEM/BEI

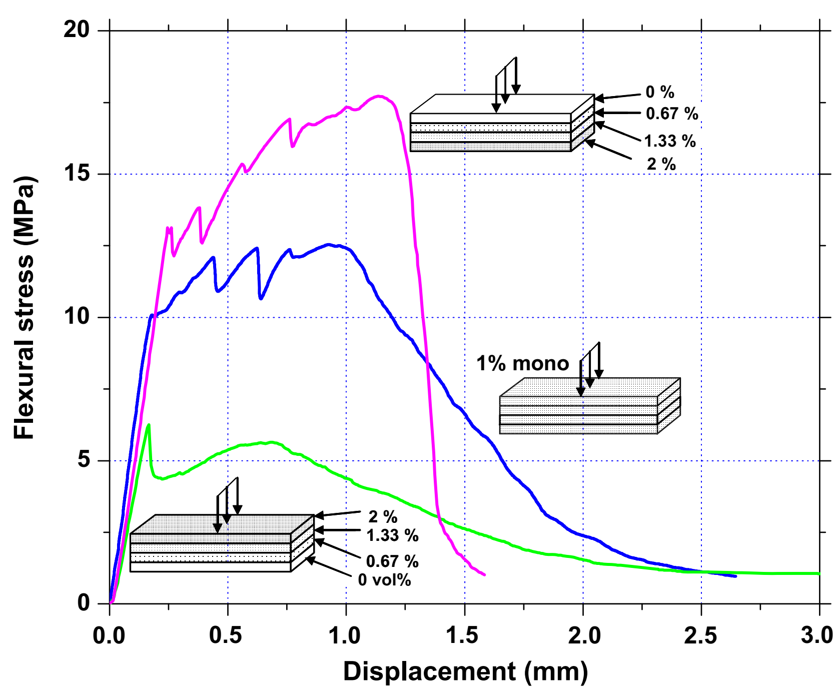

Fig. 7. Flexural response of FGFRCC with normal and reverse loading, and of homogeneous FRCC, all with the same net fiber volume fraction (1%).

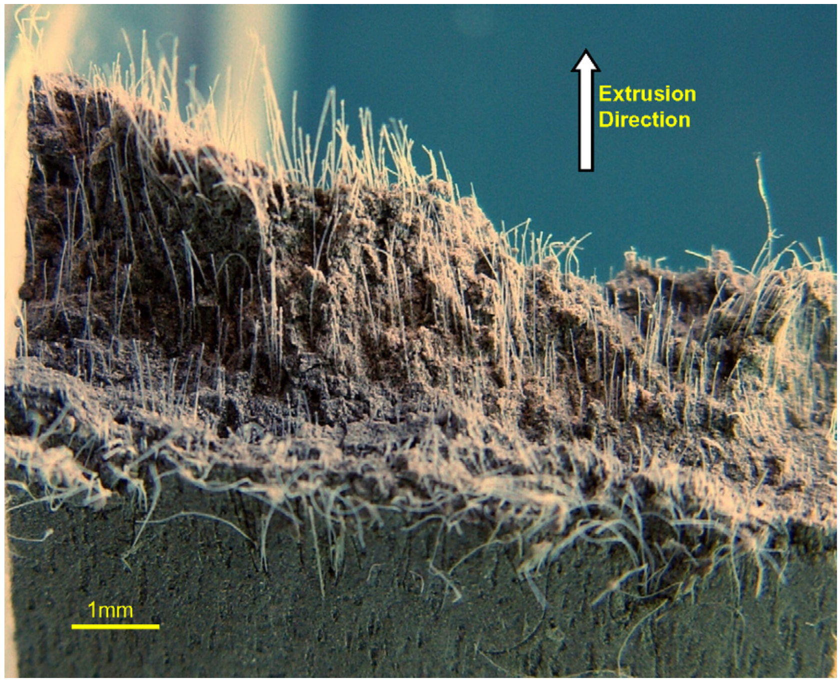

Fig. 8. Close-up optical image showing the fiber alignment in a broken specimen. Photo obtained with a Konika Minolta Dimage Z3 digital camera.

of the polished cross-section of the FGFRCC normal to the extrusion direction. From top to bottom is the direction of increasing fiber volume fraction. The small black circles are fibers, while the big, irregular gray and black patches are air voids. The continuous and more homogeneous gray area is the cementitious matrix. Fig. 9b is segmented binary image of Fig. 9a using image analysis software, showing fibers only. The image clearly shows an increase of the number of fibers from the top to the bottom. The fibers appear to be distributed randomly within each layer. Close-up views show that the cross-sections of fibers are quite circular, demonstrating parallel alignment of fibers along the extrusion direction, otherwise inclined fibers would show an elliptical cross-sectional shape [9,12]. The random distribution and parallel alignment demonstrates the effectiveness of the mixing and extrusion procedure. In Fig. 9a, no distinctive boundaries can be found between layers, the transition from one layer to the other shows seamless material transition.

Many randomly distributed and disconnected air voids are observed, mostly in the range 100–250 μm. Similar voids were also reported by Shao et al. [28] who attributed them to the use of HPMC but concluded that extruded FRCC still has higher strength than FRCC fabricated by casting.

6. Remarks on processing

The scheme used to obtain mix proportions that produce good extrudate and make pressing of FGFRCC successful is outlined in Fig. 10. We started formulating the mix proportion for the FRCC with highest fiber volume fraction, since more fibers make the FRCC more difficult to extrude successfully. Then the adjustment of other layers was relatively easy. In this way, we found through trial and error the range of mix proportions that produced satisfactory extrudates.

Matching the paste plasticity between layers during pressing is key for FGFRCC fabrication. Here the term "plasticity" is not used in a strict sense, but rather referred to the deformability of fresh FRCC layers under compression. As a result, when the plasticity was different, wavy interfaces resulted. One example is shown in Fig. 11, in which different amounts of white cement were added in three different layers for a clear visible view of the layers. The wavy interfaces between layers were due to the use of different cements, which produced different plasticity between layers. Mixes with higher fiber amounts were stiffer. The presence of fibers made the bulk fresh paste more cohesive than paste without fiber. When more fibers were added, it became more difficult to tear apart the fresh FRCC, showing increased bulk cohesiveness. However, there were some tolerances for the rheological difference between layers for pressing. One simple and effective way to control this aspect was through monitoring the total extrusion force during extrusion for each layer. Currently this is also the only parameter we could measure with accuracy. The extrusion forces for the four different layers should be similar. In the particular four layers we used to successfully fabricate the FGFRCC, we found the extrusion forces for the four layers were between 1300 and 1500 N without necessarily adjusting the water to binder ratio.

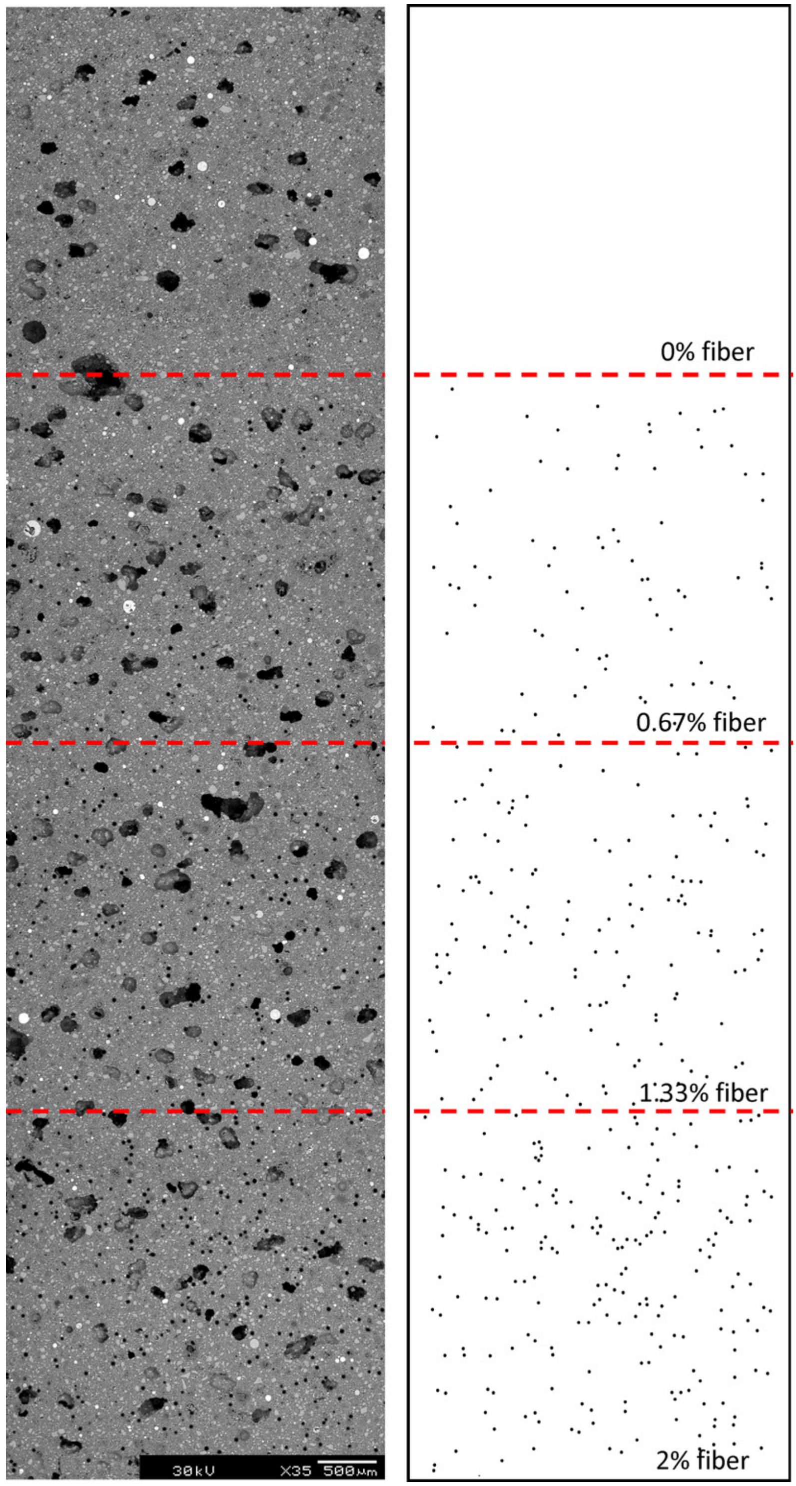

Fig. 9. FGFRCC cross-section: (a) SEM/BEI image and (b) corresponding binary image showing fiber distribution.

The FRCC layer with lower fiber volume fraction was extruded first. Before pressing, the first extruded layer had been extruded for more than half an hour. During this period of time, the agglomeration of particles and increasing action of HPMC changed the paste plasticity property, making it stiffer. This effect was utilized to help match the plasticity of later extruded layers, which were stiffer. In other words, the waiting time after extrusion of different layers is used to compensate plasticity differences between layers. This is why the four FRCC mix proportions with different fiber volume fractions can have the same water to binder ratio, yet still produce the similar plasticity for pressing.

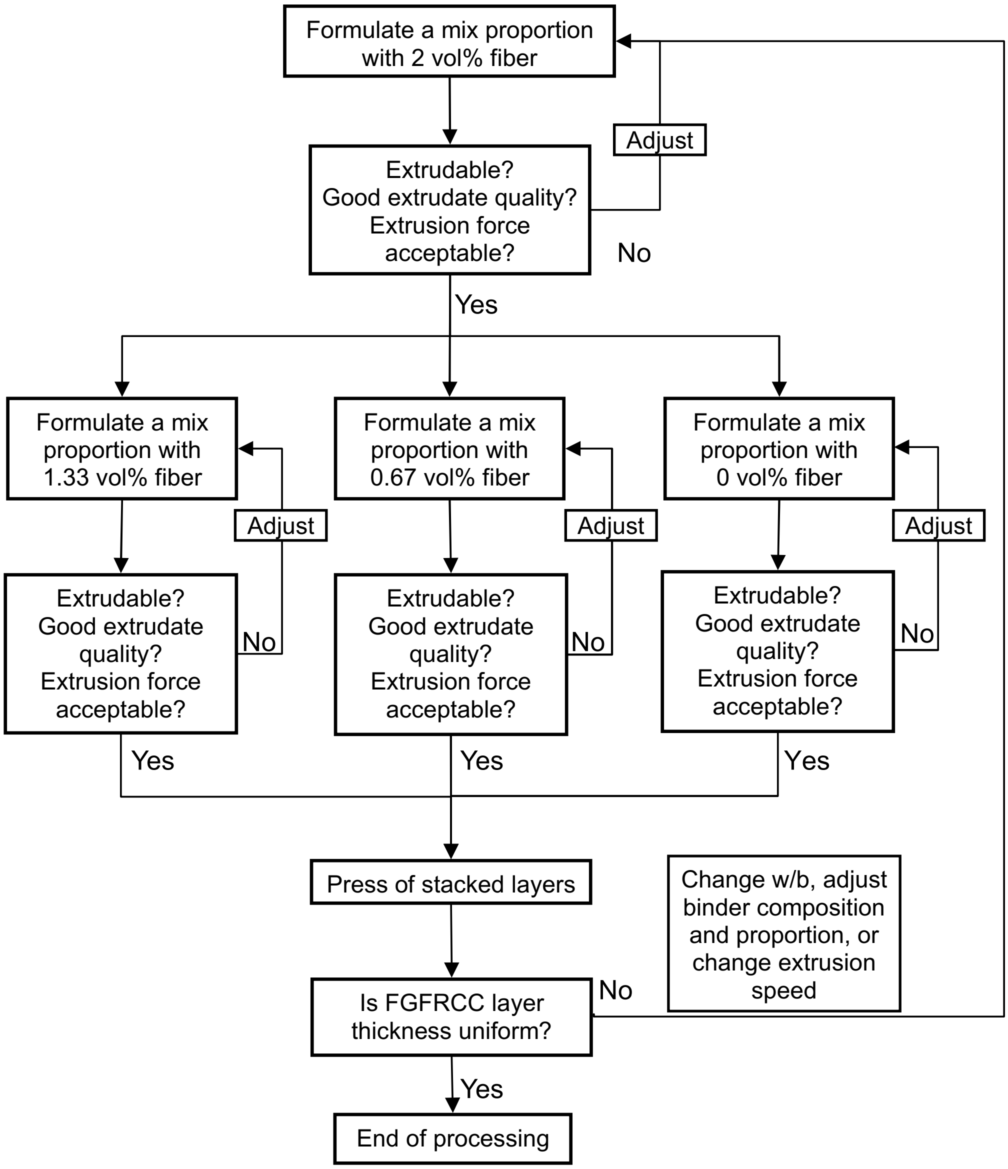

Fig. 10. Sequence of mix proportion adjustment.

Fig. 11. Wavy layers and uneven thickness resulting from inadequate control of plasticity.

7. Conclusions

Processing techniques of extrusion and pressing were used to successfully fabricate a four-layered FGFRCC. Image of the microstructure verified fiber alignment parallel to extrusion direction and graded fiber volume fractions. The FGFRCC with 1% overall fiber volume fraction showed higher strength and work of fracture compared to the homogeneous FRCC with the same fiber content. This study also indicates the relevance of proper fiber gradation. The improved properties of the FGFRCC were obtained when the compression zone had the highest fiber volume fraction, but not when a reverse gradation profile was used. It was shown that matching plasticity between layers is critical for successful pressing. When FRCC layers are not well matched their boundaries were wavy and their thicknesses were uneven.

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