Development of Self-Compacting Engineered Cementitious Composites

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Abstract

A self-compacting engineered cementitious composite (ECC) was developed by optimizing the micromechanical parameters, which control composite properties in the hardened state, and the processing parameters, which control the rheological properties in the fresh state. In the development concept of self-compacting ECC, micromechanics is adopted to properly select the matrix, fiber, and interface properties to exhibit strain hardening and multiple cracking behavior in the composites. With the selected ingredient materials, the self-compactability of ECC is then realized by the controlled rheological properties of fresh matrix and the uniform dispersion of fibers. The controlled rheological properties of fresh matrix, including deformability, flow rate, and selfcompactability is a result of adopting an optimal combination of a superplasticizer and a viscosity agent. According to the measurements of slump flow and the self-placing test result, the ECC developed in this study is proven to be self-compacting. Flexural tests demonstrate that the mechanical performance of self-compacting ECC is insensitive to the externally applied consolidation during placing. This result confirms the effectiveness of the self-compactability in maintaining the quality of the structural elements.

Keyword: self-compacting ECC, fiber, matrix, interface, micromechanics, strain hardening behavior, Hydroxypropylmethylcelluslose (HPMC)

Introduction

Engineered fiber reinforced cementitious composites (ECCs) have been developed 1,2,3 with superior ductility (about 1,000 times of the ductility of normal concrete in tension). This composite is composed of cement, sand, water, a small amount of admixtures, and an optimal amount of fibers. ECC utilizes essentially the same ingredients as most FRCs except without coarse aggregates. ECC has a tensile strain capacity of up to 6% and exhibits pseudo-strain hardening behavior, while other fiber reinforced composites (FRCs) require much larger amount of fibers for comparable mechanical properties.

Unlike some high performance FRCs, (SIFCON (slurry infiltrated 5 - 20% of steel fibers 4.5), SIMCON (slurry infiltrated 6% steel fiber mat 5; slurry infiltrated steel wool⁷), and CRC matrix (using 5 - 10% fine steel fibers⁸)), which generally adopt very high fiber contents, ECC utilizes only a limited amount of fibers. In general, only 2% or less by volume of discontinuous fibers are used in ECC. ECC also has high ultimate tensile strength (5- 10 MPa) and modulus of rupture (8-25 MPa), high fracture toughness (25-30 kJ/m²)¹ and isotropic properties. The uniqueness of ECC is not only on its tremendous mechanical properties in tension or on its relatively small amount of discontinuous fiber usage but also on its micromechanical approach in material design. This approach takes into account the interactions between fiber, matrix, and interface in the inelastic deformation process of the composite. The ECC theory provides an analytical tool for composite design without going through numerous experimental works as required by traditional trial-and-error approach in development of FRCs. Furthermore, it allows one to maximize the fiber efficiency for composite performance as well as to minimize the fiber content. The small amount of discontinuous fibers allow for flexible processing for on-site construction or off-site pre-casting as well as minimizing cost. Thus ECC is a high performance cementitious composite which embodies an advanced material design approach.

The design of ECC is based on the micromechanics that serves to establish the link between material constituents, such as matrix, fiber, and interface, and composite properties. To produce the strain hardening behavior, the micromechanical parameters are combined to satisfy a pair of criteria, the first crack stress criteria and steady state cracking criteria^{9,10,11}. The equation derived defines the critical fiber volume fraction in terms of the matrix, fiber and interface parameters.

$$V_{f} = \frac{12J_{c}}{g\tau(L_{f}/d_{f})\delta_{\text{max,bridging}}}$$
(1)

According to ECC theory, given the material parameters, the fiber content in a fiber reinforced composite must exceed this critical fiber volume fraction for the resulting

composite to exhibit strain hardening behavior. The critical fiber volume fraction is related to the material parameters shown in Equation (1). This equation also indicates that low matrix toughness and high interfacial bond are favorable to exhibiting strain hardening behavior with low fiber volume fraction. Figure 1 shows the effects of matrix toughness and interfacial bond strength on the critical fiber volume fraction as calculated based on Equation $(1)^{12}$.

In practice, insufficient vibration or consolidation of concrete usually leads to poor material quality that eventually deteriorates the durability of concrete structures. Therefore, the need for high workability or self-compactability of concrete has been recognized as a means to improve the quality and reliability of constructed facilities, especially in Japan. Self-compacting concrete requiring no consolidation works in job sites has been successfully developed and commercialized¹³.

Normal ECC processing adopts the casting method for conventional cementitious materials that generally requires high-frequency vibration to place the fresh mix into molds. The incorporation of fibers in ECC has made the placing of ECC a more challenging task than concrete. The efficiency of fibers can be significantly reduced if fibers are not uniformly distributed due to the low workability of fresh ECC mix. Thus, development of a self-compacting ECC that can flow into molds of complex figure under gravity without any consolidation is highly desirable. This self-compactability of ECC can be even more important especially when heavy reinforcement details are encountered.

To achieve self-compactability, the fresh ECC mixes should be easily deformable and should have sufficient segregation resistance. This objective may be imposed on the rheological characteristics of fresh mix, which are affected by inter-particle microstructure, fiber properties, and interfacial properties between the matrix and fibers. Specific requirements include:

- The fresh matrix should have optimal viscosity and low yield stress to provide good deformability, while preventing phase separation.
- During the casting process, the suspension should maintain its initial flowability by effective stabilization of the system.
- The flow properties of fiber-free mix should be minimally disturbed upon fiber addition to prevent poor fiber dispersion due to high fiber aspect ratio and/or high fiber volume fraction.
- The fiber surface should have optimal wettability with the fresh cementitious mix so that the fresh mix can flow homogeneously through reinforcements or complex formwork figures without any phase separation or clogging.

The objective of this work is to produce self-compacting ECC embodying the desirable properties in fresh state to satisfy the requirements above by combining flow

properties of matrix fresh mix, fiber properties, and interfacial properties, while preserving the strain hardening behavior of ECC, as indicated schematically in Figure 2. To satisfy material performance in both fresh state and hardened state, the processing parameters which affect flow properties of fresh ECC are carefully controlled in order to minimize disturbance of the micromechanical optimization for strain hardening performance of hardened ECC. Therefore, the major focus of this work is to create processing techniques which simultaneously accommodate the self-compacting and strain hardening requirements.

To quantify matrix flow properties, rheological characterization of the paste material using a rheometer is introduced. This technique provides information not only on the viscosity of material under low and high shear deformation but also on the viscosity change with time, even with small amount of sample. Thus, the matrix composition leading to the desirable matrix flow properties is determined. Based on the desirable matrix flow properties, the fresh ECC mix, in which the reinforcing fiber is dispersed, is produced.

To quantify flow properties of ECC mix, various indices, as proposed by Ozawa et al¹⁴, are adopted to characterize the rheological properties of ECC. The indices include the deformability as measured from a flow cone test and the flow rate as measured from a funnel test. These two indices basically reflect the deformability and the viscosity of a fresh mix. Some optimal magnitudes of these indices have been proposed for self-compacting concrete¹⁵. However, these optimal indices by themselves do not ensure the self-compactability of concrete. Another test using a box vessel^{13,14}, which is more realistic to the situation of practical formwork with reinforcement details, has been devised to qualify a self-compacting concrete. Therefore, a similar box vessel is also adopted in this study for a final approval of the self-compactability of ECC. In addition, the bending test of ECCs is carried out to verify the insensitivity of modulus of rupture (MOR) to the presence of external vibration, as well as the strain hardening behavior of the ECC.

Materials and Tests

Materials and mixing of fresh ECC

The ECC is made up of matrix and fibers. A high modulus polyethylene fiber (Spectra 900) with aspect ratio of 420 was used as the reinforcing fiber. The fiber

properties are given in Table 1. Ordinary Portland type I cement and silica sand were used as the major ingredients of the matrix. Silica sand was used as the fine aggregates, of which grading ranges from 0.2 to 0.3 mm. Both particles were used as received. In addition to cement and fine aggregates, various chemical admixtures were applied to control the rheological properties of fresh matrix, including melamine formaldehyde sulfate as a superplasticizer (SP) and Hydroxypropylmethylcellulose (HPMC) with molecular weight of 150,000 as a viscosity agent. The mix proportion of ECC is given in Table 2.

For the processing of fresh ECC, all of the dry solid powders were mixed in a Hobart mixer equipped with planetary rotating blade. Water was next added to form the basic matrix. Subsequently, HPMC aqueous solution was added to allow appropriate viscosity to develop in the matrix. Finally, SP was added to increase the overall workability of the matrix. The separate addition of water and SP prevents a sudden increase in the viscosity as caused by false setting¹⁶. In the last step, fibers were added manually to the cementitious matrix.

<u>Tests</u>

1. Rheological test

A Bohlin CS50 rheometer with stress control is used for the purpose of measuring rheological properties of the fresh mix. In a controlled stress rheometer, the bob rotates at a constant stress, which is above the yield stress, and the resulting strain or strain rate is measured. The viscosity of the tested matrix is then automatically calculated based on these measurements. With this rheometer, the viscosity change with time under a constant shear stress is measured to quantify the initial viscosity and the time dependence of viscosity according to changes in matrix composition.

2. Self-compactability tests

To characterize and quantify the self-compactability of fresh ECC, a number of tests were conducted, including deformability tests using slump cone or flow cone, flow rate test using a funnel device, and self-placing test using a box vessel with reinforcing bars as obstacles to ECC flow. These methods have generally been adopted to quantify the flow properties of self-compacting concrete^{14,15,17}. The geometry and dimensions of the device for each test are summarized in Table 3. The measurements of each test and the corresponding index are also given in Table 3.

Deformability test

The small flow cone for conventional flow table test without the consolidation sequence has been used to quantify the deformability of fresh mortar mix¹⁷, while the slump cone has been used to quantify that of fresh concrete mix¹⁵. In this work, both cones were used to quantify the deformability of fresh ECC.

After lifting up the slump cone, the fresh mix collapses and spreads. The maximum diameter of the spread and the diameter perpendicular to it are measured. The index for deformability, Γ_1 or Γ_2 , is then calculated based on the diameters measured as defined in Table 3.

Flow rate test

This test is conducted by filling the funnel with fresh ECC and then allowing the material to flow through the outlet freely after removing the stopper at the bottom. The time for the material to vacate from the funnel is measured. The index for flow rate, R, is calculated accordingly.

Self-placing test

A box vessel, as shown in Table 3, is prepared for this test. This box is composed of two chambers divided by a partition at the center. Three reinforcing bars with a net spacing of 1.5 cm are positioned in the gate at the bottom of the vessel connecting the two chambers. The test is conducted by filling one of the chamber with fresh ECC and removing the partition at once. Presumably, the material flows to the adjacent chamber through the gate. In order to flow through the gate, the material must pass the reinforcing bars successfully. In this study, the test device has been scaled down appropriately for ECC from that designed for self-compacting concrete¹⁵ and the net spacing of the reinforcing bars has been adjusted to 1.0 cm. As compared with the fiber length of 1.27 cm, the challenge of this test is further increased. The height reached by the in-flow ECC in another chamber was measured. An index for the degree of self-leveling is calculated by dividing this height by half of the total original height (Table 3). The index gives the value of 1 for perfect self-leveling.

3. Mechanical test

Four-point bending test with a center span of 3.2 cm was conducted to evaluate the mechanical properties of ECC. The specimen dimension is 1.27 x 7.6 x 30.5 cm. The test was conducted using a MTS testing system with 133.5 kN in total loading capacity. The specimens were loaded with a constant cross head speed in two steps. During the first 5 mm of displacement, the speed of 0.005 mm/sec was applied and then it was increased to 0.02 mm/sec. The applied load and the cross head displacement were recorded during the test.

Results and Discussion

Preparation of fresh ECC mix

Figure 2 shows a scheme to develop self-compacting ECC in the fresh state exhibiting the strain hardening behavior in the hardened state. The parameters in the left circle represent the critical factors governing the strain hardening behavior. These parameters include the mortar (matrix) toughness and the fiber bridging stress-crack opening relationship which depends on the fiber mechanical and geometric properties, fiber orientation and volume fraction, and fiber/matrix interaction parameters. Parameters on the right circle represent ones to produce self-compactability, regarding flow properties of fresh matrix mix and fiber dispersion. The factors influencing the flow properties of matrix fresh mix include W/C ratio, amount of fine aggregate, amount of admixtures such as SP and HPMC. The factors affecting the fiber dispersion include matrix flow properties, fiber geometry, fiber volume fraction, and fiber surface physicochemical properties. These parameters are combined together to satisfy the required conditions in the fresh state and hardened state.

1. Optimization of HPMC dosage

A self-compacting ECC is made possible by the desirable flow properties of matrix fresh mix, including deformability, segregation resistance, and slump loss. The rheological properties of fresh ECC mix are also strongly affected by the fiber dispersion in the matrix. Poor fiber dispersion may decrease the flowability of matrix significantly.

For the desirable rheological properties of matrix, low yield stress and optimal viscosity are required to produce high deformability, sufficient segregation resistance and uniform fiber dispersion. The solid concentration (W/C ratio), fine aggregates content (S/C ratio), and admixtures composition (polymer or fine particles) are considered significant factors affecting the flow properties. In this study, 0.3 and 0.5 are selected for the W/C ratio and the S/C ratio respectively under the guidelines of micromechanics to obtain good interfacial bond strength and optimal matrix toughness. At such a high solid concentration, the cementitious particles may form a highly flocculated network structure due to strong inter-particle attraction and the yield stress and viscosity of fresh mix may increase significantly. Therefore, it is necessary to prevent the formation of aggregated structure by reducing the inter-particle attraction in order to obtain high deformability and low slump loss of fresh mix.

Various SP have been used to improve workability in development of high performance concrete. SP neutralizes different surface charges of cement particles by adsorbing onto particles and thus dispersing the aggregates formed by electrostatic attraction. However, it has been reported that SP could fail to preserve the initial flowability with time due to the high ionic strength in dispersing medium¹⁸. This phenomenon tends to be more significant at lower W/C ratio.

HPMC has generally been used as a viscosity agent to prevent segregation of superplasticized cementitious suspension of high W/C ratio ranging from 0.5 to 0.6^{19} . In this work, HPMC is incorporated into the matrix to preserve initial flowability of mix at low w/c ratio. At the optimal concentration, HPMC can prevent flocculation among particles without increasing the overall viscosity due to the excluded volume effect. Besides, it can also prevent segregation caused by particle collision.

Figure 3 shows the effect of HPMC concentration on the time dependence of viscosity of the fresh cement paste. In this test, cement particles were mixed with water at a W/C ratio of 0.3. HPMC concentration varies from 0.0% to 0.05% by weight of cement. The SP concentration was kept constant at 4% which is the saturated dosage for the viscosity. A constant shear stress of 25Pa, which is slightly higher than yield stress of fresh matrix, was applied and the resulting shear rate was measured. This plot shows that the viscosity of fresh mix increases with time, regardless of changes in polymer concentration, as the rate of built-up of flocculated structure exceeds the rate of plastically deforming structure under the applied shear stress. At various HPMC concentrations except 0.05%, the measurements of initial viscosity of fresh mix are comparable. The fresh mix without HPMC shows a monotonous increase in viscosity. However, the fresh mix dispersed by 0.013% of HPMC shows a decrease in initial viscosity followed by a slow increase. The initial decrease in viscosity is interpreted as the breakdown of weakly flocculated structure formed during the sample loading. The rate of viscosity growth with time is higher at HPMC concentration above 0.025%. The highest rate of viscosity growth occurs at 0.050% of HPMC.

From the viscosity data described above, it can be concluded that the cementitious suspension without HPMC or with high HPMC concentration induces the strongly flocculated structure resistant to shear deformation, while the suspension with optimal HPMC concentration prevents it. This study suggests that the HPMC dosage of 0.013% results in an optimal deformability in the cement paste. When fine aggregates are added to the cement paste, the deformability is expected to be optimal as well with this HPMC concentration. This has been verified by conducting deformability test of the fresh mortar. The test result indicates that the mortar with 0.013% of HPMC and 4% of SP gave the maximum diameter, 26cm, among all others. The calculated deformability, 5.5, is considered to be appropriate to make qualified self-compacting concrete¹⁷.

2. Self-compactability of ECC

In development of self-compacting concrete, the amount of coarse aggregate has to be optimized to minimize the friction between aggregates that increases the resistance to concrete flow. In addition, at the optimal content, coarse aggregates may contribute to enhance the self-compactability by increasing the unit weight. On the other hand, the low density (0.97 g/cm³) of the PE fiber used for ECC causes reduction in the unit weight of

ECC and thus decrease the driving force for ECC to flow. Therefore, the original flow properties of matrix mix can be greatly reduced. Further hampering of the flow properties are the potential entanglement and poor wettability.

In order to make ECC self-compacting, the above negative effects in the addition of fiber against ECC flow should be minimized, so that the deformability of fresh matrix can be preserved as much as possible. Therefore, using short fiber and limited fiber volume fraction are necessary to ensure uniform fiber dispersion and to minimize the reduction in matrix deformability. In this study, Spectra fiber with 12.5 mm in fiber length and 1% of fiber volume fraction was selected for development of self-compacting ECC.

Table 4 summarizes the results from self-compactability tests and a number of derived indices of the self-compacting ECC. The results for ECC were measurements in the present study, while those for mortar or concrete are adopted from the literature for references. According to Table 4, the deformability of matrix for ECC (Γ_1), 5.76, exceeds the criteria for mortar¹⁷. The deformability of ECC (Γ_2), 8, is acceptable based on the standard of self-compacting concrete¹⁵. The flow rate of ECC (R), 1.67, is higher than those proposed or optimal values of mortar or self-compacting concrete¹⁷, revealing ECC can flow faster than self-compacting concrete does. In order to verify the self-compactability of ECC in practical placing situation, the self-placing test was conducted by allowing ECC to flow through a gate of reinforcing bars with net spacing simply comparable to the fiber length. The resulting height reached by ECC, 13 cm out of 30 cm in original height, is considered to be qualified for self-compacting based on the standard of self-compacting concrete¹⁵.

Figure 4(a) shows that the ECC fresh mix spread up to 60cm uniformly after the slump cone was lifted up. Figure 4(b) shows the ECC fresh mix streaming through the outlet of the funnel from the bottom without any segregation or clogging. It took 6 seconds to empty the funnel. Figure 4(c) shows that the fresh mix was able to flow from the right chamber through the reinforcing bars under the partition and rise up to a surface level of a remarkable height of 13 cm in the left chamber.

Mechanical Properties

In order to study the mechanical properties of the self-compacting ECC developed in this study, a number of specimens for flexural tests were cast using ECC materials after a series of self-compactability tests. The specimens were cast with and without applying external high-frequency vibration during placing.

Figure 5 shows that the strain hardening behavior of self-compacting ECC is observed and the flexural strength and strain capacity do not depend on the presence or absence of external compaction process. In fact, the test result shows slightly lower

modulus of rupture (MOR) for the specimens with vibration applied. This could be attributed to the possible phase separation in a fresh mix caused by vibration. Figure 6(a) shows the tested specimen with the appearance of multiple cracking, although in remarkable deflection, can still take the bending load. Figure 6(b) shows a uniform cracking pattern on the tension zone of the specimen. The result confirms the ECC material developed in this study exhibit strain hardening behavior successfully.

Conclusion

In this study, by optimizing micromechanical parameters and processing parameters, self-compacting ECC has been successfully developed. This self-compacting ECC shows not only the self-compactability in fresh state and but also the strain hardening behavior in hardened state. All the flow indices obtained from fresh ECC mix are comparable to those of self-compacting concrete. The self-compactability of ECC is further confirmed by the self-placing test. The desirable flow properties of fresh matrix is realized by incorporation of optimal combination of HPMC and SP and of optimal fiber length and fiber content in compromise with requirements for hardened mechanical properties. The four point bending test confirms that the strain hardening behavior and strength of composite material are insensitive to the presence or absence of the external consolidation process using the self-compacting ECC developed in this research.

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Table 1 Dimensions and mechanical properties of Polyethylene fiber

Diameter (mm)	Length (mm)	Strength (MPa)	Elastic Modulus (GPa)	Density (g/cm³)
0.038	12	2700	120	0.98

Table 2 Mix proportion of self-compacting ECC (in weight ratio)

Cement	Sand	Water	НРМС	SP	Fiber (volume fraction)
1.0	0.5	0.30	0.013	0.04	0.01

Table 3 Tests and devices

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Test	Deformability test I	Deformability test II	Flow rate test	Self-placing test
	(Flow table test)	(Slump flow test)	(Funnel test)	(Box vessel test)
	Flow cone with 10 cm in bottom diameter and 6 cm in height	Slump cone with 20 cm in bottom diameter and 30 cm in height	Funnel with a 3 × 3 cm outlet and cm ³ in total volume	2-chamber vessel with max. height of 30 cm and 12 mm-rebars in 2.5 cm of spacing at the connection
Dimensions of test device	7 cm 6 cm	30 cm 20 cm (d _O)	3 cm	30 cm (h ₀)
Type of measurement	Diameter of the spread, \overline{d}_2 (cm)	Diameter of the spread, \overline{d}_1	Time for material to vacate from the outlet, \bar{t} (sec.)	Height reached by the inflow material, h
Corresponding index	Deformability, Γ_I	Deformability, Γ_2	Relative flow rate, R	Degree of self-leveling, L
Conversion	$rac{ar{d}_2 - ar{d}_0}{ar{d}_0}$	$rac{\overline{d}_1 - \overline{d}_0}{\overline{d}_0}$	$\frac{10}{t}$	$\frac{\overline{h}}{(h_0/2)}$
Note	Standard test for fresh mortar except no consolidation is applied	Standard test for quality control of fresh concrete	Dimensions of funnel proposed by Ozawa et al for mortar test	Dimensions scaled down for ECC

Table 4 Results of self-compactability tests

		Proposed or optimal values for self-compactability		
Material	ECC	Mortar ^{13,15,17}	Concrete ^{13,14,15}	
\bar{d}_1 (cm)	18(26)*			
Γ_1	2.24(5.76)*	5		
\bar{d}_2 (cm)	60		60 - 72	
Γ_2	8	-	8 - 12	
\bar{t} (sec)	6			
R	1.67	1	0.8 - 1.22	
\bar{h} (cm)	13			
L	0.87	1	1(0.73) [@]	
Notes	* () denotes matrix properties		()denotes the criterion proposed by Nagamoto and Ozawa	

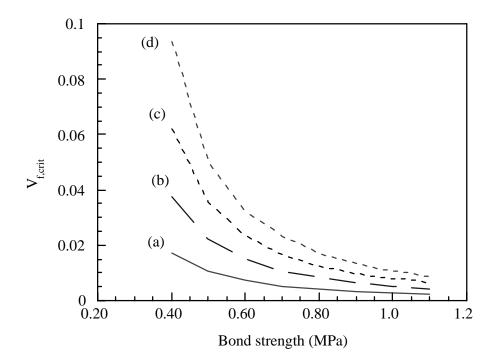


Figure 1 Effect of matrix toughness and interfacial bonding strength on the critical fiber volume fraction (E $_{\rm f}$ = 120 GPa, $L_{\rm f}$ = 12.7 mm, $d_{\rm f}$ = 0.038 mm, g = 2.0, $E_{\rm m}$ = 25 GPa) (a) J_c = 0.005 kJ/m² (b) J_c = 0.010 kJ/m² (c) J_c = 0.015 kJ/m² (d) J_c = 0.020 kJ/m²

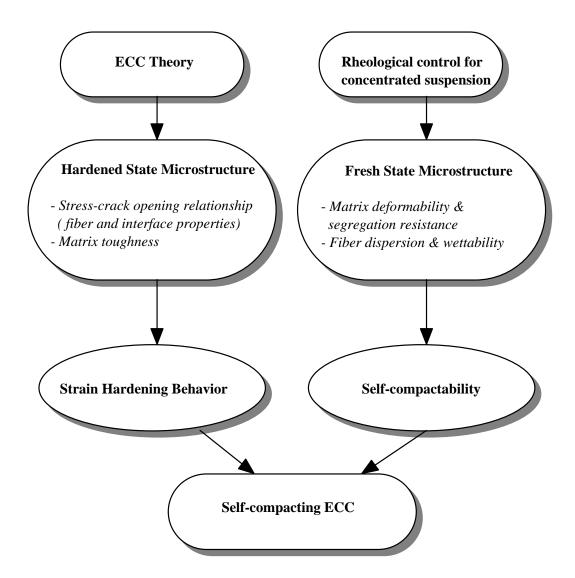


Figure 2 Scheme for the development of self-compacting ECC by combining self-flowable fresh properties and micromechanical design

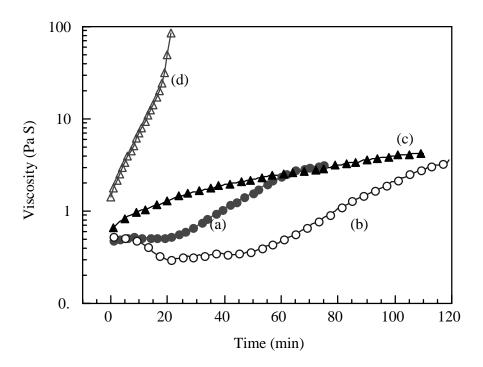
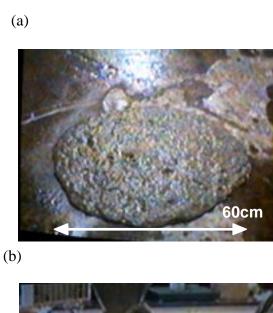


Figure 3 Viscosity change with time of fresh cement paste as a function of HPMC concentration (W/C=0.3, SP Conc. = 4%) (a) No HPMC (b) HPMC 0.013% (c) HPMC 0.025% (d) HPMC 0.050%





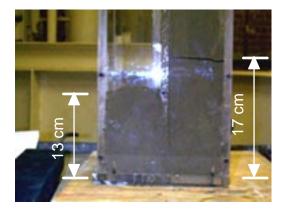


Figure 4 Visualization of flow properties of self-compacting-ECC (a) highly deformed ECC fresh mix by gravity, (b) good flowability of fresh mix through the exit at the funnel bottom (c) Self-compactability to flow through the reinforced bars in a box

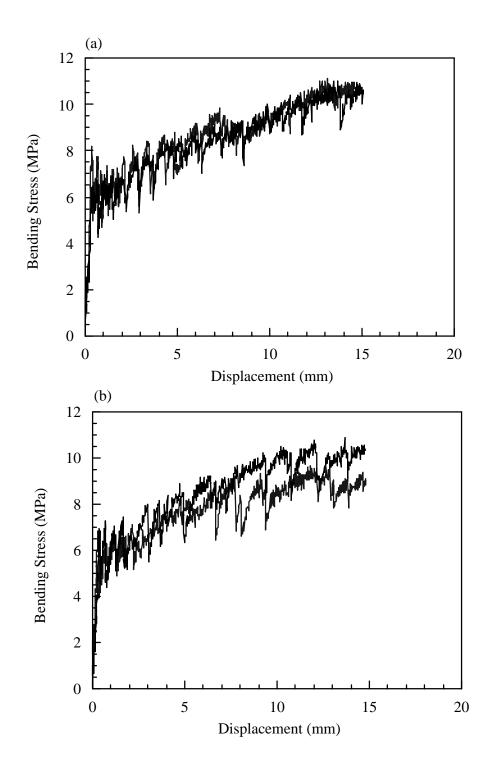
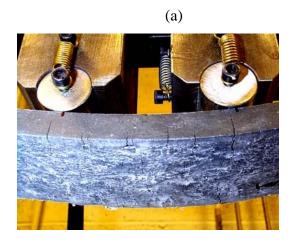


Figure 5 Bending test result of self-compacting ECC (a) No external compaction , MOR = 11.1, 11.0 MPa (b) Application of external compaction, MOR = 10.9, 9.7 MPa



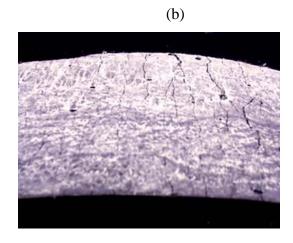


Figure 6 Crack pattern of bending specimen (a) during load (b) at maximum load