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Scientific paper

Experimental Study on Self-Healing Capability of FRCC Using Different Types of Synthetic Fibers

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Abstract

Experimental studies are carried out to evaluate the self-healing capability of FRCC using different types of synthetic fibers that have different chemical properties, i.e. poly vinyl alcohol (PVA), ethylene vinyl alcohol (EVOH), polyacetal (POM) and polypropylene (PP). FRCC specimens were subjected to tension tests in order to generate a crack, and the cracked specimens were immersed in water. In order to evaluate the effect of self-healing phenomena, permeability tests and microscopic observation were carried out. Microscopic observation revealed that the high polarity of synthetic composite has high potential of self-healing precipitation around fibers bridging a crack. Moreover, the coefficient of water permeability was generally reduced with this chemical precipitation, especially in the PVA series. However, even when it was confirmed by microscopic observation that precipitation had appeared and filled a crack, we found there is no recovery of water tightness in some cases, i.e. in the EVOH and POM series. It is confirmed that not only the chemical properties of fibers but also the geometrical properties of the crack surface, such as roughness, complexity and continuity, affect the capability of self-healing for water tightness.

1. Introduction

To maintain the global environment and build a sustainable society, highly durable and long-life concrete structures are strongly required. However, cracking is inherent in concrete structures and it leads to serious damage that cause durability issues, e.g. increase in permeability, ingress of aggressive agent and corrosion of reinforcing steel. It is important to perform periodic and/or detailed inspection, and implement proper repairing procedures for preventing such damage and deterioration. On the other hand, even in the case of ordinary concrete, cracks with small width of 0.1 mm or less tend to close autogenously in the presence of moisture due to precipitation of calcium carbonate (Edvardsen 1999). A number of studies have been carried out to enhance the self-healing capability of concrete using several approaches. Reinhardt and Joose (2003) revealed that curing temperature has a great influence on self-healing of cracked concrete. Ahn and Kishi (2010) applied some types of mineral admixtures and Jonkers *et al.* (2010) demonstrated quite a novel approach using bacteria as a self-healing agent in order to enhance self-healing capability. Such crack closing phenomena are useful for preventing invasion of aggressive agent,

though efficient recovery of strength is doubtful.

In Japan, a technical committee of the Japan Concrete Institute (JCI) summarized recent research on self-healing concrete, giving definitions of various types of such concrete (Igarashi *et al.* 2009). Per these definitions, enhancing methods that use admixtures are categorized as “autonomic healing,” i.e. involuntary healing of cracks by admixtures. Fiber reinforced cementitious composite (FRCC) is one of the autonomic healing materials. FRCC has mechanical characteristics that can control crack propagation in the cement matrix through bridging with short fibers. Since the self-healing process requires crack widths that are sufficiently small, FRCCs are expected to have great self-healing capability. Li *et al.* (1998) carried out experimental studies on the self-healing capability of Engineered Cementitious Composites (ECC) and concluded that cementitious materials with inherently tight crack width effectively self-heal. Kan *et al.* (2010) identified self-healing products of ECC as mainly C-S-H and calcium carbonate through ESEM-EDS and TEM observation. Qian *et al.* (2009) showed the self-healing behavior of fiber reinforced strain hardening cementitious composites incorporating blast furnace slag and limestone powder. The recovery in deflection capacity and stiffness was found to range between 65% and 105% in virgin specimens, significantly higher compared to the specimens without benefit of the self-healing process. Mihashi *et al.* (2011) declared FRCC to have better resistance against corrosion of steel bars than mortar because of the fiber bridging of cracks and the self-healing of some of the cracks. Homma *et al.* (2009) confirmed that fibers bridging the crack surface play a role not only as a crack width controller but also as a core for depositing chemical products, and that a larger number of bridging

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fibers achieves more effective self-healing. Koda *et al.* (2011) concluded that different types of fibers show different levels of self-healing performance. In particular, fibers with polarity (e.g. PVA) promote more effectively the deposition of crystallization products than other types of fibers.

In this study, the self-healing capability of FRCCs containing synthetic fibers with different chemical properties is investigated to reveal the role of the fibers as a core for the precipitation of calcium carbonate and to enhance the potential of the self-healing function of FRCC.

2. Self-healing mechanism

Self-healing is a natural process of crack repair that can occur in concrete in the presence of moisture. The deposition of calcium carbonate is said to occur as the result of the following reactions (Edvardsen 1999).

The dissolved CO_2 ion and the calcium ion Ca^{2+} in water combine with each other to produce calcium carbonate crystals, and the resulting calcium carbonate crystallization is precipitated on the crack surface. As a result, the crack width is gradually reduced and ultimately the crack closes. Homma (2009) revealed also that the self-healing mechanism was accelerated in the crack with bridging fiber. Polymers that have polarity can attract Ca^{2+} and become a template of calcium carbonate (Kato *et al.* 2008). For that reason, some types of synthetic fibers, such as PVA, can attract Ca^{2+} and promote the deposition of crystallization products.

3. Testing procedures

In this study, two experiments (hereafter called A and B) were carried out to evaluate the self-healing capability of FRCC using different types of synthetic fibers. In experiment A, the volume of the precipitated crystals of calcium carbonate around each fiber that was exposed from the crack surface of FRCC plates was measured to

estimate the potential of self-healing. In experiment B, microscopic observation of the crack surface and a water permeability test on FRCC plates containing different types of fibers were carried out to evaluate the self-healing capability.

3.1 Experiment A (observation of precipitated crystals around each fiber)

The precipitation of crystals of calcium carbonate around each fiber exposed from the crack surface of FRCC immersed in curing water was observed and the volume of the precipitation was measured. **Table 1** lists the mix proportion of the FRCC specimens used in experiment A and experiment B. **Tables 2** and **3** gives the properties of the employed synthetic fibers and the other materials, respectively. **Figure 1** shows the chemical constitution of the fibers, with the circles indicating polarity groups. Polarity strengths depend on electro-negativity, which is the tendency of an atom or a functional group to attract electrons. In **Fig. 1**, PVA has the highest polarity strength due to the OH radical, and PP has no polarity strength. EVOH consist of both PVA and PP parts, and POM has the O radical. Thus, the polarity strengths of EVOH and POM are of moderate level between PVA and PP. Prismatic specimens of 40 mm × 40 mm × 160 mm were prepared. After standard curing for 7 days, each specimen was subjected to the 3-point bending test and completely separated. A half piece of specimen was cut into

Table 1 Mix proportion of FRCC specimens.

W/B	S/B	SF/B	SP/B	Fiber (Vol. %)
0.45	0.45	0.15	0.009	2.0

B: binder (cement + silica fume)

Table 3 Properties of employed materials.

Material	Symbol	Properties
Cement	C	High early strength Portland cement Density: 3.14 g/cm ³
Silica fume	SF	Density: 2.20 g/cm ³
Sand	S	Silica sand #5, Density: 2.61 g/cm ³ , Diameter: ~500μm
Super plasticizer	SP	Polycarboxylic acid ether system Density: 1.05 g/cm ³

Table 2 Properties of employed fibers.

Series	Type of Fiber	Density [g/cm ³]	Tensile Strength [N/mm ²]	Length [mm]	Diameter [μm]	Shape of Section
PP	Polypropylene	0.91	760	6	11	Circle
C-PP					18	Deformed
POM	Polyacetal	1.41	135	10	48	Circle
EVOH	Ethylene vinyl alcohol copolymer	1.04	231	5	15	Circle
PVA-I	Polyvinyl alcohol	1.30	1600	6	14	Circle
PVA-II					37

Fig. 1 Characteristic parts of chemical constitution.

Fig. 2 Testing procedure of experiment A.

30 mm height, and the broken surface was observed under microscope. Next, it was immersed in a water tank of 150 cc volume. **Figure 2** shows the schematic diagram of the testing procedure. Following immersion of the specimens in curing water for 7 days, fibers exposed from the broken surface were observed and the diameter of each fiber was measured using a digital microscope. **Figure 2 (c)** shows a sample of fiber covered by the precipitated crystals. The precipitation thickness was calculated from the difference between the measured whole diameter and the original diameter of the fiber. Here, the catalogue data was used as the original diameter because minimal scatter was confirmed by microscopic observation. Measurements were carried out over 15 points for all fibers projecting from the broken surface, and the thickness was calculated as the mean value.

3.2 Experiment B (evaluation of self-healing capability of FRCCs containing different synthetic fibers)

In experiment B, self-healing capability was confirmed from morphological change of chemical products precipitated around cracks obtained by microscopic observation and from the coefficient of water permeability obtained by water permeability test. **Figure 3** shows a specimen and the apparatus employed for the uniaxial tension test. The geometry of the specimen is a plate measuring 85 mm $\times$ 85 mm $\times$ 30 mm fixed in place with 4 screw bars (M6) with an anchor nut at the tip (Homma

2009). The same materials and mix proportion as those used in experiment A (**Tables 1, 2 and 3**) were employed. In the first curing step, the specimens were settled in a

Fig. 3 Apparatus and specimen of experiment B.

Fig. 4 Apparatus for water permeability test (Homma *et al.* 2009 and Kishimoto *et al.* 2007).

Table 4 Maximum crack width evaluated by means of microscopic observation (μm).

Specimen No.	Series					
	PP	C-PP	POM	EVOH	PVA-I	PVA-II
No. 1	240	301	350	287	423	225
No. 2	248	176	361	399	327	185
No. 3	250	195	367	339	—	106

water tank of 20°C temperature and 50 cm water depth for 6 days after demolding. After the first curing step, the specimens were subjected to a uniaxial tension test using 4 embedded screw bars to generate a crack approximately 300 μm wide. Metal plates were settled through the installed nuts on both sides of the specimen to maintain this crack width (Fig. 3). The maximum crack width of each specimen that was evaluated by means of microscopic observation is shown in Table 4. Except for the crack width, the three specimens were used under identical conditions. In the case of multiple cracking, the widest crack width of the main crack was measured as the maximum crack width, because the maximum crack width could be considered as a more sensitive parameter than average crack width for FRCC specimens featuring multiple cracks in order to evaluate the coefficient of water permeability (Nishiwaki 2004).

The cracked specimens were subjected to a water permeability test. Figure 4 shows the apparatus of this permeability test. This testing apparatus was developed from the method used by Kishimoto (2007) and Homma (2009). The coefficient of water permeability was calculated from the volume of permeating water through the

specimen. After the test, as the second curing step, the specimens were immersed under the same conditions as the first curing step. During this second curing step, the water permeability test was carried out again after 3, 14 and 28 days. At the same time, the surface around the crack was observed under digital microscope. After curing for 28 days (at the end of the second curing step), the specimens were subjected to the uniaxial tension test again, and the fractured surface of the specimens was observed under digital microscope to obtain information on the precipitation area and of the geometry of the crack surface.

4. Results and discussion

4.1 Experiment A

Figure 5 shows the average thickness of precipitated crystals of calcium carbonate on different types of fibers. According to this figure, the smallest precipitation was observed in the PP series. The precipitation thickness increased in the order of the POM, EVOH, PVA-I and PVA-II series. This tendency corresponds with the strength of polarity that was estimated from the chemical formula shown in Fig. 1. The polarity of the synthetic composite, e.g. hydroxide ions, suggests that the positively charged calcium ions are attracted by a strong electric field (Manoli, 2002). Therefore, a polarized fiber tends to attract Ca^{2+} ions and precipitate calcium carbonate. While the type of fibers used in the PVA-I and PVA-II series was the same but the diameters of the fibers differed between the two series, the fibers showed almost the same quantity of precipitated chemical products. Thus, it was revealed that the diameter of the fibers

Fig. 5 Average thickness of precipitated chemical products.

has a slight influence on precipitation. Therefore, it is expected that thinner fibers with the same volume content can produce a larger number of cores on fibers bridging the crack than thicker ones, and that they can

supply a larger total quantity of self-healing precipitation in the crack. However, the tendency was different in the case of the C-PP and PP series, where a larger quantity of precipitation was measured in the former series than in the latter series. It was confirmed that fibers whose section is more complex and has a longer perimeter can provide a larger quantity of self-healing products than fibers whose section is of simple geometry.

4.2 Experiment B

4.2.1 Uniaxial tension test

Figure 6 shows the relationship between tensile stress and elongation under the uni-axial tension test carried out for crack generation at the age of 7 days. According to this figure, the tensile strength of all the series was 2 to 3 N/mm², and ductility depends on the type of fiber. The PP, C-PP, PVA-I and PVA-II series showed ductile behavior even under uniaxial tensile stress. After the tension test, the residual crack width could be controlled to be approximately 300 μm. However, the POM and EVOH series showed brittle behavior and the specimens were almost instantaneously split into two parts just after the

(a) PP series

(b) C-PP series

(c) C-PP series

(d) EVOH series

(e) PVA-I series

(f) PVA-II series

Fig. 6 Relationship between tensile stress and elongation in first loading test to generate a crack.

peak load. The brittle behavior was due to the slight bond strength between the fibers and cement matrix, i.e. the bridging effect was not sufficient to obtain a ductile behavior. Therefore the unloaded specimens were set in the condition of a crack width of approximately 300 μm using metal plates with embedded nuts and bolts.

4.2.2 Microscopic observation

Figure 7 shows the surface of each specimen including the maximum crack width obtained by microscopic observation. Just after the loading test (for crack generation), the edges of the crack could be clearly recognized. However, after water curing, self-healing products ap-

peared and specimens containing different types of fibers showed different patterns of crack filling product precipitation. Concerning the PP series, crystallization products could be found only around the bridging fibers and they filled up only a small part of the crack even in the case of 14-day curing. In the other series, the crack of all the specimens was filled with precipitated crystals after 14-day curing. In both of the PVA series, remarkable self-healing was observed even after only 3 days of curing. These tendencies agree with the results of experiment A as discussed in section 4.1. In the case of ordinary plain concrete, the maximum crack width that can be filled by natural self-healing phenomena is under

		Days of the second curing step (days)		
		0	3	14
Series (Original maximum crack width)	PP-3 (250 μm)
	C-PP-1 (301 μm)
	POM-2 (361 μm)
	EVOH-2 (399 μm)
	PVA-I-2 (327 μm)
	PVA-II-1 (225 μm)

Fig. 7 Microscopic observation of precipitated products around crack.

0.1 mm (Edvardsen 1999). That is to say, FRCCs with synthetic fibers have greater self-healing capability than ordinary concrete.

4.2.3 Water permeability test

Figure 8 shows the relationship between curing days in the second curing step and the coefficient of water permeability (k). The k value was calculated by equation (4).

$$k = \frac{Q \cdot l}{t \cdot A \cdot h} \quad (4)$$

where Q = quantity of water flow [m^3], l = thickness of the specimen [m^2], t = time [sec], A = surface area of the plate specimen [m^2] and h = pressure head [m] (here, 0.135 m).

In Fig. 8, the k value of the specimen without any crack is shown by dotted lines. Figure 9 (a) shows the normalized coefficient of water permeability in the case of the PP, C-PP, PVA-I and PVA-II series, which showed relatively ductile behavior under uniaxial tensile stress. The normalized coefficient of water permeability was estimated as the ratio of the value of k in each curing day

Fig. 8 Relationship between time and coefficient of water permeability.

Fig. 9 Relationship between time and average normalized coefficient of water permeability.

to the k value just after crack induction. As a result of self-healing phenomena, that is, filling of the crack with precipitated products, water permeability was reduced and water tightness was recovered. In the case of the PP series, however, the reduction of the coefficient of water permeability was not confirmed. By contrast, the C-PP specimen recovered its water tightness during the second curing step. In the cases of the PVA-I and PVA-II series, the coefficient of water permeability was remarkably reduced up to approximately 0.1 to 10×10^{-11} (m/s), which approximates that of specimens without any cracks (approximately 0.7×10^{-11} m/s). That is to say, taking the self-healing capability into account, FRCC using PVA fibers can allow a crack width of at least 0.3 mm, which is much larger than the 0.1 mm width allowed in the case of ordinary concrete. Moreover, the PVA-I series reduced the coefficient of water permeability more effectively than the PVA-II series. This is due to the increase in the number of bridging fibers across the crack because PVA-I is much thinner than PVA-II. The employed type of synthetic fiber obviously affected the

water tightness recovery ratio result.

Figures 8 (c) and (d) show the relationship between the number of days of the second curing and the coefficient of water permeability of the POM and EVOH series. Figure 9 (b) shows the relationship between time and the average normalized coefficient in the case of the POM and EVOH series, which showed brittle behavior in the tension test (see Figs 6 (c) and (d)). In both of these series, the coefficient of water permeability was not reduced by the self-healing phenomena, though self-healing products were observed under microscope as discussed in section 4.2.2. That is to say, self-healing products around the crack of the specimen cannot guarantee the recovery of water tightness. Since these series showed brittle behavior in the tension test, we considered that not only the chemical properties of the employed fibers but also the geometric properties of the crack surface affected the capability of self-healing. In the following section, the geometry of the crack surface was observed as one of the aspects of the FRCC specimen related to the mechanical properties.

4.2.4 Observation of the fractured surface of the specimen

After 28 days, that is the end of the second curing step, a uniaxial tension test was carried out again and the specimens were completely separated. Figure 10 shows detailed views of crack surfaces. Self-healing products remaining on the surface were observed, because the crack generated by the first loading was grown again in the same place by the second loading. The hexagonal shapes in the section are the marks left by the anchor nuts placed at the tip of the screw bars (Fig. 3). In almost all the specimens, the generated crack was located in the section including the anchor nuts. Self-healing products were found around the marginal area in all the series. However, self-healing products were observed in the center area of the crack surface only in the PVAs series. That indicates that the PVA series is more effective in creating self-healing products, including inside the crack.

As aspects of cracking properties, roughness and tortuosity of the crack surface were observed under digital microscope. Figure 11 shows the observed area and measured lines, which correspond to the data depicted in Fig. 13. The microscope employed in this study (VHX-1000, Keyence Corporation) has a function to automatically measure the depth of the surface three dimensionally. The depth is calculated based on the locations that are in (and out of) focus in obtained photographs (Nayar 1994). Figure 12 shows an example of the obtained contour map of a crack surface. From this figure, the surface area was automatically calculated by the digital microscope. Figure 13 shows the roughness of the crack surface measured along the five lines in Fig. 11. The PP and PVA-I series, which showed ductile behavior in the tension test, had rougher and more complex surfaces than the POM and EVOH series, which showed brittle behavior in the tension test. Moreover, Fig. 14

Fig. 10 Self-healing products on surface of crack.

shows the ratio of the crack surface area to the reference base area ($25 \times 35 = 875 \text{ mm}^2$). This figure clearly shows that the brittle series (POM and EVOH) had a smaller crack surface area and simpler surface geometry than the others series. On the other hand, the specimens of the ductile series (PPs and PVAs) had a complex and rougher surface, and the PVAs series in particular had the most complex crack surface geometry.

The water-flow rate through a crack in concrete is described as a function of the cube of the crack width per the following formula (Tsukamoto *et al*, 1991):

$$q = \alpha \frac{g I \ell w^3}{12\nu} \quad (5)$$

where q = water flow in concrete cracks [m^3/s], α = em-

Fig. 11 Measured lines for roughness of crack surface.

Fig. 12 Calculated surface area and reference base area.

Fig. 13 Roughness of crack surface.

pirical parameter [-], g = gravity acceleration [m/s^2], I = pressure gradient (h/L), h = height of fluid column on the inlet side [m], L = crack length in the flow direction [m], ℓ = crack length at a right angle to the flow direction [m], w = crack width [m], and ν = kinematic viscosity [m^2/s].

Here, we can recognize α as a coefficient related to the

Fig. 14 Ratio of crack surface to reference base area.

Fig. 15 Relationship between ratio of crack surface to reference base area and coefficient of water permeability before self-healing.

geometry of the crack surface differing from a smooth plate, e.g. roughness of crack surface, crack branching, and accumulation of microcracks around the main crack. Thus, α is related to the ratio shown in **Fig. 14**. In this study, the crack width of the specimens was adjusted to approximately 300 μm . Before the self-healing process, the coefficients of water permeability k and α are related to each other (**Fig. 15**). Homma *et al.* (2009) demonstrated that more complex crack surfaces have greater capability of self-healing. Here, the ductile series with larger α (PVAs) reduced the water permeability more effectively than the brittle series (EVOH and POM). The specimens of the ductile series tended to have multiple cracks and crack branches while the specimens of the brittle series were separated into two pieces with a single crack. Such complex and discontinuous cracks have a fracture process zone at the tip, where self-healing products can be easily deposited because of their rather narrow crack width. However, the PPs series, which had mediate level α , had lower capability of self-healing because of lack of polarity of reinforcing fibers. Namely, both of the ductility and chemical properties were required in order to enhance the capability of self-healing

Fig. 16 Relationship between normalized coefficient of water permeability, thickness of self-healing precipitation and ratio of crack surface to reference base area.

of FRCC. These results are summarized in **Fig. 16**, which shows the relationship between the capability of self-healing (the normalized coefficient of water permeability), the polarity of the employed fibers (the thickness of self-healing precipitation), and the complexity of the geometry of the crack surface (the ratio of the crack surface to the reference base area).

Based on the findings of the present study, it is suggested that selecting the optimal type of fiber most effective for self-healing of FRCC should be done from the viewpoint of not only the potential of chemical precipitation but also the cracking properties of the whole FRCC material.

5. Concluding remarks

This paper presents the results of experimental studies on the self-healing capability of FRCC using different types of synthetic fibers. Based on the results, the following conclusions can be drawn:

- (1) FRCC using synthetic fibers have a great self-healing capability, because synthetic fibers not

only control crack width but also serve as a core of the chemical precipitation site. In particular, synthetic fibers with high polarity can promote effective precipitation of self-healing products.

- (2) The PVAs series, which have the highest polarity, can recover water tightness by self-healing phenomena for crack widths up to 0.3 mm.
- (3) It is confirmed that not only the chemical properties of fibers but also the geometrical properties of the crack surface, i.e. roughness, complexity and continuity, affect the capability of self-healing for water tightness.

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