VERIFICATION OF SELF-HEALING MECHANISM IN OPC INCORPORATING CARBONATE

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ABSTRACT

In concrete structures, tiny cracks have great effects on the point of durability, and self-healing concrete can heal these cracks. In authors' research group, the self-healing concrete incorporating inorganic materials has been developed. Although the self-healing ability has been proved practically, the effects of each self-healing materials and whole mechanism of self-healing are not clear enough. Therefore, the objective of this study is to verify the mechanism of self-healing. Especially, the abilities of unreacted cement and carbonates are focused. The phenomenon in pure OPC and cement materials containing carbonates are observed in both experimental and practical conditions. The first experiment is simple soaking test and water-pass test. The result suggests the pure OPC specimens in young ages shows the self-healing ability by producing calcium carbonate, and premixed carbonates affects this phenomenon. And this ability of OPC is diminished by time but there is possibility that some carbonates can maintain the self-healing ability. The second experiment is the chemical analysis of the cores pulled out from the self-healed specimens using SEM-EDS and XRF. By this observation, the appearance and composition of crack-filling materials are affected by the proportion of cement paste and aggregate around cracks. This study suggests that production of crack-filling materials such as calcium carbonate is greatly affected by the amount of unreacted cement and premixed carbonates.

Keywords: Style, format, checklist, Microsoft Word, EASEC13.

1. INTRODUCTION

Recently, the deterioration of concrete structure become apparent and cracks of concrete deteriorate the function, durability and appearance in the structure. To solve these problems, the ability of healing crack process in concrete has been paid attention, and the self-healing concrete are developed(Tae-Ho Ahn et al. 2; Koide et al. 2012; Morita et al. 2011).

In previous research, A lot of kinds of addition agents which produce self-healing ability have been developed, but the process of self-healing has not been disclosed enough. In our research group, the self-healing concrete using mineral admixtures has been developed (Koide et al. 2012; Morita et al. 2011).

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Table 1: Mixing Compornent

Water-pass test

name	air (%)	W/C %	W kg/m^3	C kg/m^3	S kg/m^3	NG/LG kg/m^3	SP kg/m^3
N55	4.5	55	168	306	806	1007	2.4
N49	4.5	49	171	349	802	953	1.4

Box specimen for obeservation (Morita 2011)

name	air (%)	W/C %	W kg/m^3	C kg/m^3	S kg/m^3	NG/LG kg/m^3	SP kg/m^3
plain	4.5	57.9	168	290	826	1019	0.8

※ 50% of NG/LG is limestone

Table 2: Curing condition

Curing method	Temperature ℃	RH %	
Air	20	60	91days in air
Chamber	20(air)→60(chamber)	60(air)→80%(chamber)	7days in air →16days in chamber

In these researches, although the specimens of self-healing concrete show self-ability greatly, the OPC specimens also healed its cracks partly. This result suggests the OPC potentially have the self-healing ability. It is not obvious how much healing ability OPC can have and how addition agents can affects. In order to improve the self-healing concrete, the whole healing process must be revealed and measure the effects of each self-healing agents.

Therefore, the objective of this study is to verify the mechanism of self-healing. Especially, the abilities of unreacted cement and carbonates are focused. The phenomenon in pure OPC and cement materials containing carbonates are observed in both experimental and practical conditions.

2. EXPERIMENT

2.1. Water-pass test and TG-DTA

In order to estimate self-healing ability, water-pass test is operated. Specimens are cylinders which diameter is 100mm and height is 200mm. After specific curing period, artificial cracks are introduced from top to bottom. In this experiment, self-healing ability is estimated by the water speed go through this artificial crack.

The mix proportion of specimens is shown in table 1. W/C ratio is 49% and 55%, and Ordinal Portland cement is used. The Curing data is shown in table 2. The sealed curing and accelerated curing is used. In past research, the self-healing ability of OPC is produced by unreacted cement, so the accelerated curing can decrease the self-healing ability. For acceleration, after sealing curing in 7days, specimens are put in chamber for 16 days. In the chamber, the temperature is 60℃ and the

Fig. 1: Water-pass test

Fig. 2: Box specimens(Morita 2011)

related humidity is 80%. Acceleration of curing can be calculated by the equivalent age which is 91 days.

To make artificial crack, specimens are split from two notches on side. After cleaning the split face and put Teflon tape, which is 0.2 mm thick, on the face, they are banded again. On the surface, the width of artificial cracks is balanced to be 0.2-0.4 mm.

In the water-pass test, specimens are hold in flowing water situation, shown in Fig. 1. On the top, they are set the plastic tube. Water is kept in this tube and flow through the cracks continuously. The flow speed is measured after 1,3,7,14,28 days. From the changing process of flow speed, the healing ability is estimated.

The amount of water reacted with cement is measured by TG-DTA. The degree of hydration can be estimated by this result, the cement hydration Specimen used for TG-DTA is cement paste with 49% W/C ratio. The curing method is two types, sealing curing and accelerated curing, based on the water-pass test. After curing, pastes are broke into particles and soaked in acetone for 1hour to stop hydration. For drying acetone, they are put in vacuum for 24 hours

2.2. Observation of self-healing products

Self-healing products are observed by Microscope and SEM-EDS. For this observation, the specimen of past research

Morita et al. 2011) is used. It was casted in March 2010, and water pass test was done. The form is hollow cube shown in Fig. 2.

The mixing component is shown in Table 1. The aggregate consists of 50 part normal aggregate and 50 part limestone aggregate. From this specimen, one core is extinct and cut in round slices after solidification using epoxy. After polishing, cutting plane of core is observed by Microscope and SEM-EDS

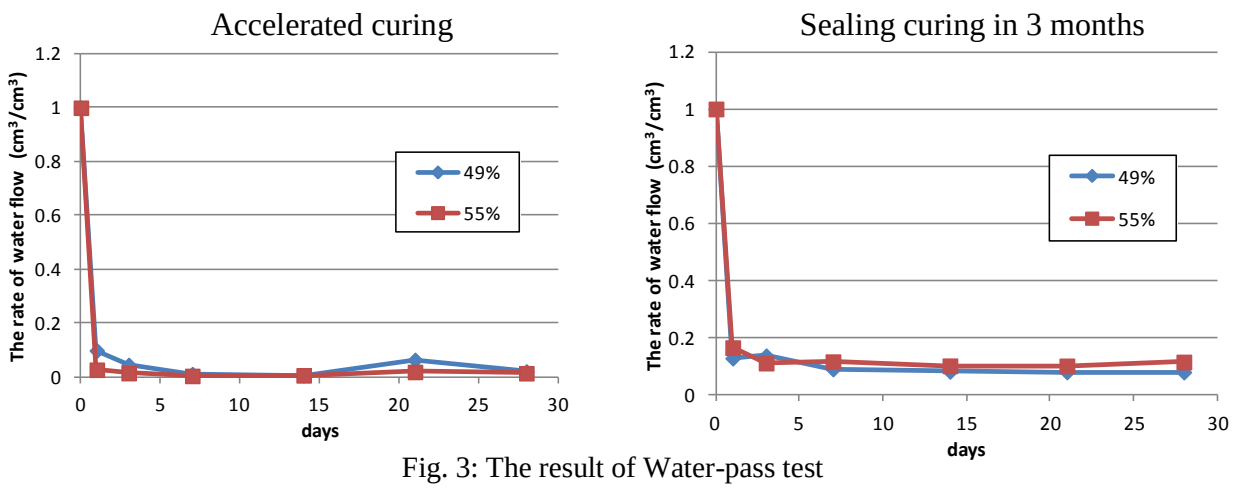

Fig. 3: The result of Water-pass test

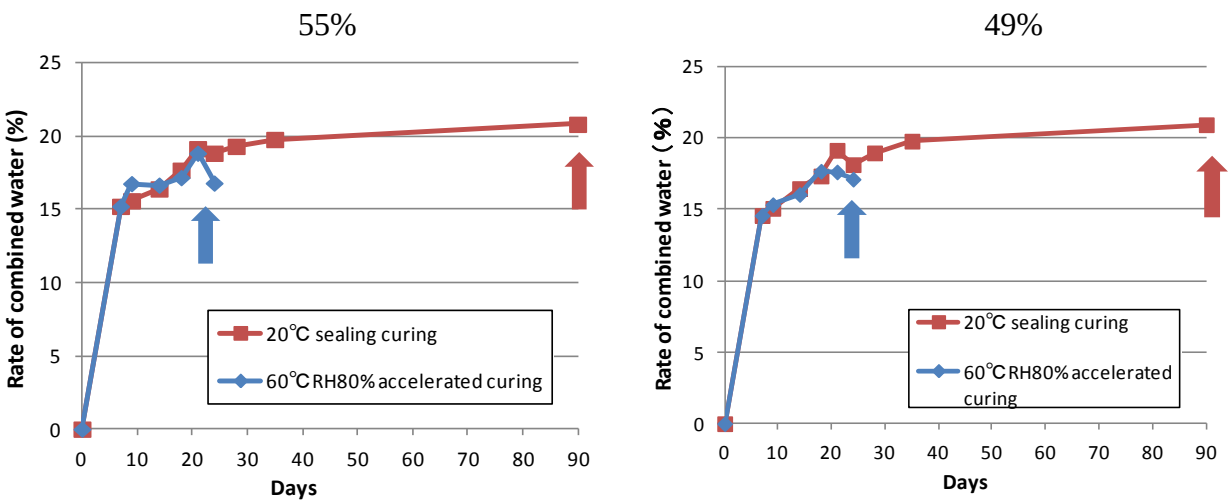

Fig. 4: The result of TG-DTA

The past research found this specimen has great self-healing ability. This observation focuses on the aggregate. In this specimen, 50% of the aggregate consists of limestone, which is kind of carbonate. It is supposed that the carbonates in the concrete can improve the production of carbonates. Hence, products around aggregate are selectively observed.

3. RESULT

3.1. Water-pass test and TG-DTA

The result of water-pass test is shown in Fig.3. Each data is average of 3 specimens. The vertical axis represents the rate of water flow, which is the ratio of water flow speed of each measurement to the initial speed. Although both 49% and 55% W/C ratio specimen has almost same tendency, difference of curing method affects the result. After accelerated curing, water flow is almost stopped. However, after sealing curing in 3 months, water flow is not stopped. Fig.2 shows the self-healing products. Specimens in accelerated curing have much self-healing products.

To confirm the main cause of self-healing ability of OPC, the hydration rate is measured by TG-DTA. In TG-DTA, the hydration rate is represented by the amount of water reacted with

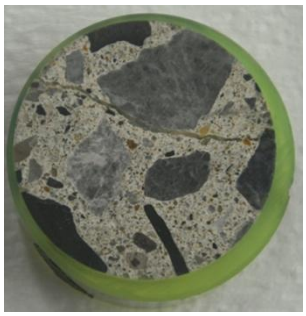

Fig. 4: Cutting face of Core

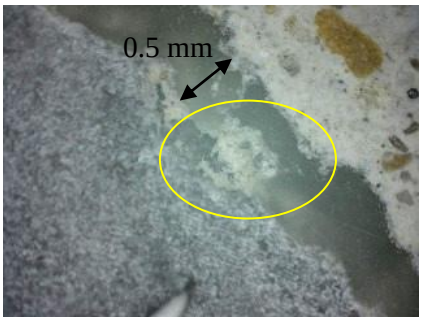

Fig. 5-(a)

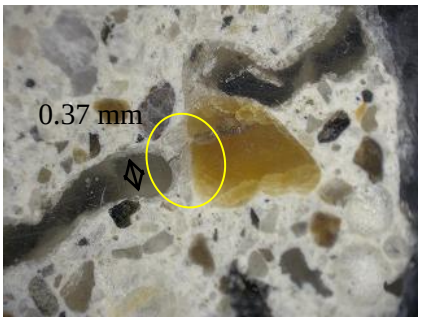

Fig. 5-(b)

Fig. 5: Observation by Microscope

Element	Weight %
Si	22.622237
Ca	77.377763

(a)

Element	Weight %
Mg	13.91
Si	22.95
Ca	40.99
Fe	22.15

(b)

Fig. 7: EDS system

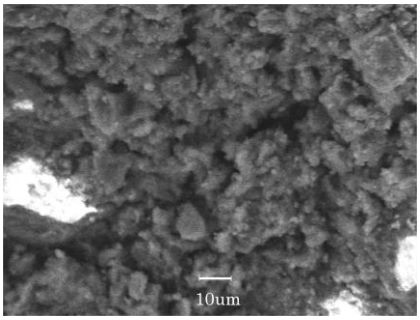

Fig. 6-(a)

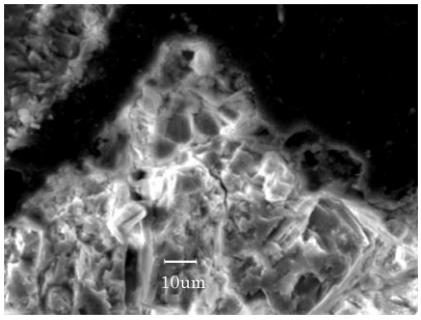

Fig. 6-(b)

Fig. 6: Observation by SEM-EDS

cement. The result is shown in Fig 4. The water-pass test is done in the arrow point. In the accelerated curing specimen, hydration does not proceed as expected. Besides the same effective

age, the specimen with accelerate curing has less reacted water than that with sealing curing. In other words, the accelerated curing used in this paper does not work well to accelerate cement hydration. It is supposed that the starting period of accelerate curing is too late. Once the hydration reaches to be the state of rate-limiting by time, the temperature does not affect the hydration. In this research, the accelerated curing begins after 7days, which is too late to accelerate hydration.

The result of water-pass test can be explained by the unreacted cement. Because the specimen with accelerated curing has more unreacted cement than expected, self-healing ability is maintained. In contrast, because the specimens with sealing curing do not have enough unreacted cement, they do not have self-healing ability. Hence, the unreacted cement is supposed to maintain self-healing ability.

3.2. Observation by Microscope and SEM-EDS

The observation of self-healing products is conducted using Microscope and SEM-EDS. The cutting plane of the specimen core is shown in the Fig.4. In the core, crack face and products in the crack are solidified by epoxy. First, By Microscope, the crack face is observed to find self-healing products. Secondly, products are observed and analyzed by SEM-EDS to identify the crystal component.

Fig. 5 shows the self-healing products around the aggregate. The curving point of crack around aggregates is the place where the white products are often found, such as Fig 5-(a). However, products are sometimes found around the strait line of crack facing aggregate, such as Fig. 5-(b).

Then, the products are observed by SEM-EDS, shown in Fig.6. From these pictures, the crystal form of each product is different. In other words, it is each product has different generating process and different components.

The further analysis by EDS system substantiate this supposition. Fig 7 shows the result of EDS system. From this result shows the product shown in Fig6-(a) are consist of Calcium products. It is speculated that they are made by calcium silicate hydrates. However, the products shown in Fig 6-(b) consist of Calcium and Magnesium. Although cement does not contain Magnesium, Limestone aggregate often contain Magnesium as dolomite. Therefore products are produced from aggregate, especially limestone aggregate.

As a result, it can be insisted that kinds of self-healing products and their producing process have multiple ways and not only cement but also especially aggregate can be the factor of self-healing ability.

4. CONCLUSION

From this research, two facts below can be concluded.

1. Unreacted cement can affect the self-healing phenomenon. During the early period after casting, OPC specimen has self-healing ability. However, with hydration process, the extinction of unreacted cement eliminates the self-healing ability.

2. The observation shows the possibility that aggregate, especially limestone aggregate, help to make self-healing products.

In the Future research, new self-healing concrete will be developed which encourages the self-healing ability of OPC.

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