

**投稿論文 (英文)**  
**PAPERS**

# SIGNIFICANCE OF IRON LAYER AS AN INDICATOR TO DETERMINE THE MICROBIAL CORROSION OF CONCRETES

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Determination of microbial corrosion is very important to anticipate the remaining service life of the concrete structures in order to avoid the recurrence of structures collapse or failures. The behaviors of Fe-ions and formation of Fe-rich layer were studied. Fe-ions started to migrate to carbonated zone during carbonation process. When surface pH decreased by microbial corrosion process, Fe-ions accumulated and concentrated locally forming Fe-layer at areas between corroded and sound zones. Results show that Fe-layer depth is more than the depth of corrosion. Calculation of Fe-layer formation rates as a mean to determine the corrosion rate with certain factor of safety is proposed.

**Key Words :** microbial corrosion, concrete, indicator, *T.thiooxidans*, iron layer, ICP

## 1. INTRODUCTION

The microbial corrosion of concrete sewer pipes and wastewater treatment facilities have caused severe damages and incur substantial amount of budget when comes for construction and rehabilitation works. Since the forties, microbial corrosion phenomena has been considered a serious problem not only in warm climate but also in countries with moderate climate<sup>1)~9)</sup>. Eventhough our understandings about microbial corrosion have increased during the past years, some questions concerning the deterioration process of concrete and structural failures remained unsolved.

Sulfuric acid is produced by oxidation of hydrogen sulfide gas by *Thiobacillus thiooxidans* on the moist surface of concrete. Sulfuric acid attack progresses slowly, destroys the concrete and reacts with steel reinforcement causing pipe to loose its structural strength and collapse. If the pipe is located under the road pavement, it will pose not only a safety hazard but also prevent the smooth flowing in the pipe due to fall of debris *etc.* The failures of these concrete structures mostly were due not to flaw of the designs or construction practises, but due to inability to anticipate the performance and remaining life of concrete structures in service. Therefore, controlling the corrosion process and accurate determination of

rate of microbial corrosion are necessary for sewer authorities to prepare maintenance and replacement plannings.

Several properties and indicators have been established to acknowledge the investigators about the degree of deterioration process and possible failures due to sulfuric acid attack. Engineering indicators include the change in length, mass loss, thickness, compressive strength, flexural strength, or modulus of elasticity. Chemical manifestations as result of sulfuric acid attack include the increase of sulfur content<sup>7)</sup>, formation of gypsum, ettringite<sup>8)</sup>, and pH level. Precise corrosion quantification was well correlated microbiologically between cell numbers of *T.thiooxidans* and grade of corrosion<sup>9)</sup>.

Review about microbial corrosion of concrete has been dealt with length by Mori *et al.*<sup>9)</sup>. Among others, the importance of Fe-layer in typical corroded concrete in determination of microbial corrosion rate was highlighted. Our previous and current studies show that besides the physical and chemical changes and subsequent formation of corrosion products, concrete subject to sulfuric acid attack is also characterized by the formation of iron layer in corroded samples. Iron layer with thickness of 100~200  $\mu\text{m}$  was first identified by Tazaki *et al.*<sup>10)</sup> at areas between sound and corroded concretes. Beside sulfur and other elements, the present of Fe (iron) element was also detected in corroded samples through microscopic studies<sup>7),11)</sup>, but no explanation was given. Physical appearance of corroded concretes under attack by sulfuric acid experience discoloration and display red or brown staining due to dissolution of iron. The main source of iron in concrete structures is from the cement compound. In ordinary Portland cement, tetracal-

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cium aluminoferrite ( $C_4AF$ ) presents in oxide form as  $Fe_2O_3$  is one of the main compounds of cement. It constitutes 0.5~6.0 percent of weight of typical Portland cement<sup>12)</sup>.

Accurate prediction of corrosion rate is very important to avoid the recurrence of structures collapse and failures. Based on this approach, this paper attempts to investigate the movement of Fe-ions and subsequent formation of Fe-layer during microbial corrosion. Results obtained will be used to evaluate its potentials as an indicator to determine the rate of microbial corrosion.

## 2. MATERIALS AND METHODS

### (1) Carbonation and Microbial Corrosion Experiments

Mortar specimens measuring  $40 \times 40 \times 160$  mm were prepared by mix proportion of 520 g cement (OPC), 1040 g Toyora standard sand, and 330 g water. The specimens were cured at 100% RH for one day and subsequent curing in water for 27 days at  $20^\circ C$ . Then, for three months the specimens were stored in humid box at  $20^\circ C$  before the initiation of the experiment. Mortar specimens were exposed to three different  $CO_2$  gas concentrations of 5%, 0.5%, and control for four months. Then, the specimens were inoculated with *T.thiooxidans* with exposure to 100 ppm  $H_2S$  gas and respective  $CO_2$  concentrations for about eight months period. The experimental set-up of the laboratory experiment is shown in Fig.1. The details were given in another paper<sup>13)</sup>.

In order to investigate the actual condition, core samplings were conducted in three small treatment plants. Core samples of concrete structures on-site were collected by core sampler as shown in Fig.2. Surface pH of concrete was determined by pH paper. Depth of carbonation was measured by spraying the concrete with 1% ethanol-phenolphthalein solution.  $H_2S$  and  $CO_2$  concentrations were measured by using  $H_2S$  meter (GASTECH H-S 1050) and  $CO_2$  test tubes (GASTECH No.2L & 2LL), respectively.

### (2) Elemental Analysis of Carbonated and Corroded Samples by ICPS (2000)

After breaking the specimens and sprayed 1% phenolphthalein solution, three regions from the specimens exposed to  $CO_2$  gas were selected; carbonated zone, boundary of carbonated-sound, and sound zone (pink-colored zone). In case of microbial corroded mortar specimens, samples from four zones were selected; surface, Fe-layer, carbonated, and sound (Fig.3).

Samples were crushed into powder manually or by vibrating mixer mill. The fine powder was obtained by sieving through  $210 \mu m$  opening mesh

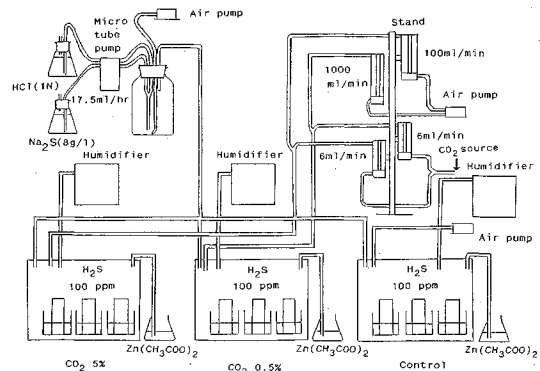


Fig.1 Set-up of carbonation and microbial corrosion experiment.

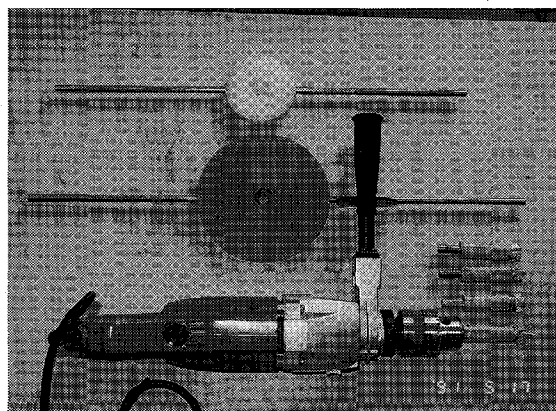


Fig.2 Concrete core sampler apparatus.

70. 50 mg of powdered sample was placed into the Teflon vessel and was added with 0.5 ml hydrofluoric acid and 1 ml HCl. After sealing the vessel and leaving it to stand for more than 16 hours at room temperature, the contents were transferred to a poly-vinyl bottle with 6 ml 4% boric acid ( $H_3B$ ). After an hour, the extracts were diluted with distilled water to 100 gram. Then, finally the extracts were filtered by filter paper and ready for use for ICP analysis (ICPS 2000). To prepare for standard solutions, 50 mg of the following standard rocks were dissolved according to the same method as above : JR-2 Rhyolite ; JA-1 Andesite, JB-3 Basalt, JB-2 Basalt, JGb-1 Gabbro, and blank. Blank solution was prepared by mixing 1 ml 12 N HCl with 6 ml Boric acid and 0.5 ml HFl and weigh to 100 g with distilled water.

## 3. RESULTS AND DISCUSSION

### (1) Carbonation

Results of ICP analysis of specimens exposed to three different concentrations are shown in Table 1. The concentration of Fe-ions in carbonated, boundary of carbonated-sound, and sound showed

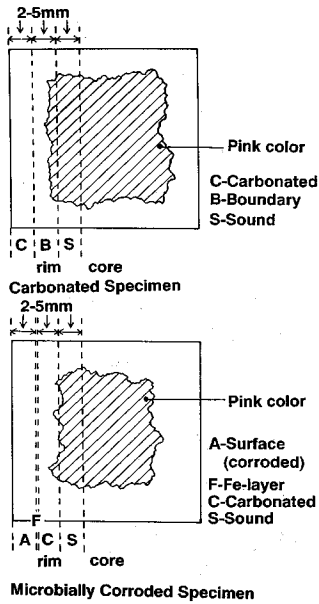


Fig.3 Sampling for ICP analysis.

a tendency to increase indicating the movement of Fe-ions from inside to outward direction during carbonation. These results were confirmed by the previous report<sup>14)</sup> that the cations (positive ion charge) migrate from inside to surface during carbonation process. Specimens exposed to 0.5% CO<sub>2</sub> gas experienced gradual and consistent increased of Fe-ions concentration at all zones. Reduction of pH at surface and successive layers by neutralising effect of carbonation was responsible for the continue migration of Fe-ions to the outer layers.

(2) Microbial Corrosion

When carbonated specimens began to experience the microbial corrosion attack, surface pH of specimens decreased and sulfuric acid began attacking the deeper layers. Discoloration of specimens surface has started to occur after about one month where surface pH were about 4.0~4.5. Kong and Orbison<sup>15)</sup> reported that specimens immersed in pH 2 of sulfuric/nitric acid solutions exhibited color change within the first 48 hours of immersion. Generally, in all specimens, degree of discoloration increased as surface pH decreased. 0.5% specimens exhibited faster discoloration but at end of the experiment, both 0.5% and control specimens displayed wider brownish surface area.

Breaking the specimens at sewage level revealed the formation of very thin Fe-layer line formed between the carbonated zone and corroded surface (Fig.4). Fe-ions concentration measured by ICP at carbonated zone continued to increase at faster rate as result of microbial corrosion (Table 2).

Table 1 The concentration of Fe-ions during carbonation process.

Carbonation Period (days)	CO <sub>2</sub> Concentration	C <sup>1)</sup>	Fe (%) B <sup>2)</sup>	S <sup>3)</sup>	Average (%)
32	Control	0.59	0.53	0.51	0.54
55		0.57	0.57	0.62	0.59
96		0.49	0.51	0.45	0.48
120		1.44	0.78	0.88	1.03
32	0.5%	0.55	0.50	0.58	0.54
55		0.64	0.60	0.64	0.63
96		0.70	0.91	0.66	0.76
120		0.79	1.03	0.86	0.88
32	5%	0.62	0.49	0.56	0.56
55		0.56	0.73	0.87	0.72
96		0.87	0.73	0.70	0.76
120		0.74	0.78	0.89	0.80

<sup>1)</sup>Carbonation <sup>2)</sup>Boundary Carbonated-Sound <sup>3)</sup>Sound

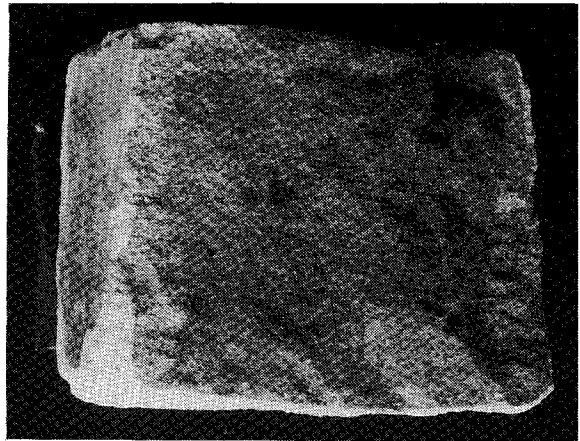


Fig.4 Fe-layer between carbonated zone and corroded surface.

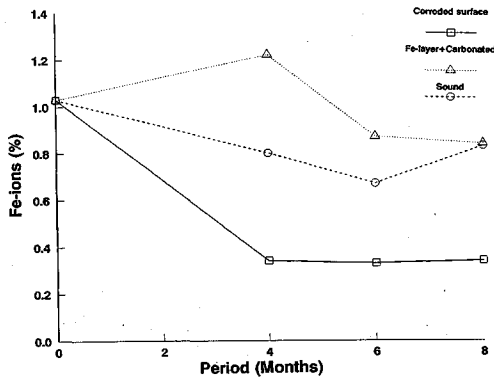
Table 2 The concentration of Fe-ions at different zones during microbial corrosion process.

Specimens	Zones	Fe-ion Concentration (%)		
		4 Months	6 Months	8 Months
Control	Surface	0.34	0.33	0.34
	Around Fe-layer	0.87	1.07	0.93
	Carbonated	1.57	0.66	0.75
	Sound	0.80	0.67	0.83
0.5%	Surface	0.40	0.59	0.45
	Around Fe-layer	0.80	2.47	1.33
	Carbonated	0.88	0.63	0.78
	Sound	0.80	0.69	0.74
5%	Surface	0.71	0.44	0.63
	Around Fe-layer	0.91	0.94	1.41
	Carbonated	1.49	2.79	1.22
	Sound	0.78	0.66	0.81

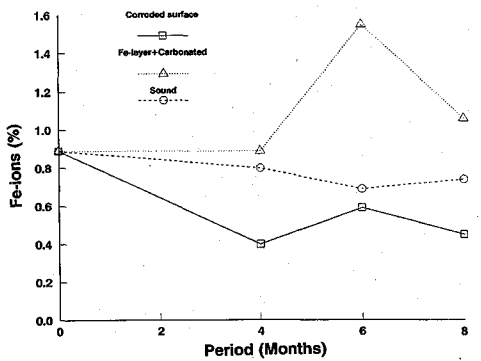
\* Samples were collected near the sewage level

After four months from the initiation of microbial corrosion process, Fe-ions measured at carbonated zones in 5%, 0.5%, and control specimens increased from 0.74, 0.79, and 1.44 to 1.49, 0.88, and 1.57 percent, respectively (Tables 1, 2). Surface pH drop from about 8.0 after four months carbonation to about 1~2 caused an increase of 101, 11.4, and 9.0 percent in 5%, 0.5%, and control, respectively. Discoloration of specimens showed brownish and red stain at areas above water level where pH was very low.

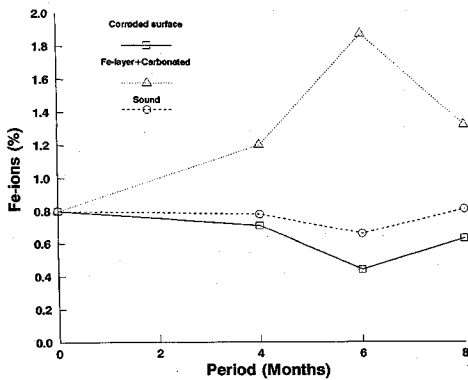
By microbial corrosion process, Fe-ions gradually accumulated and concentrated locally forming very thin iron layer with pH range between acidic-alkalic<sup>10)</sup>. As a result, the concentration of Fe-ions in Fe-layer increased with time in contrast with a



(a) Control



(b) 0.5% CO<sub>2</sub>



(c) 5% CO<sub>2</sub>

Fig.5 Change of Fe-ions concentration during microbial corrosion processes.

decrease in corroded zone (Fig.5). The results indicated that the rate of Fe-ions movement depend on pH of the surface and attacked layers which behaved in correspond with the microbial corrosion process. 0.5% specimens had the highest carbonation rate followed by 5% and control specimens. Referring to Table 2, Fe-ions concentration in Fe-layer zone of 0.5% specimens has started to exceed the carbonated zone after four

months from the beginning of microbial corrosion process. In 5% and control specimens however, Fe-ions concentration in Fe-layer began to exceed the carbonated zone after six and eight months, respectively. Higher degree of pH in the later and formation of much calcite in the former which provided resistance to sulfuric acid penetration were assumed responsible for the slow migration of Fe-ions causing delay in attaining concentrated Fe-ions in Fe-layer zone.

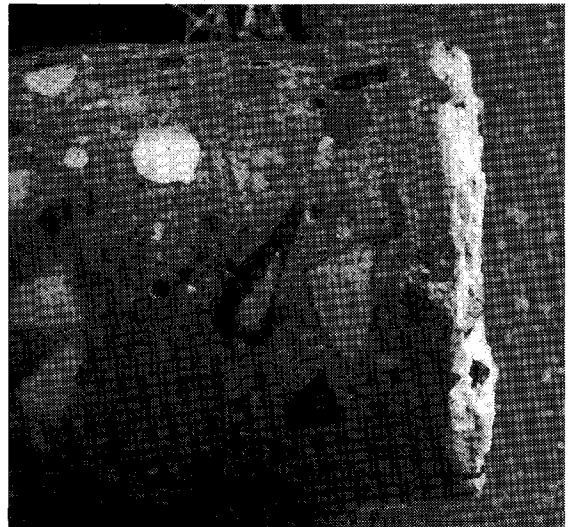
At this stage, determination of bivalent and trivalent irons (Fe<sup>2+</sup> and Fe<sup>3+</sup>) have been analyzed by Mössbauer. The spectrum of the corrosion sample at room temperature shows that there are at least two forms of iron present. The spectral parameters of doublet I indicate that it is due to Fe<sup>2+</sup> while the parameters of doublet II indicate the presence of Fe<sup>3+</sup>. This spectrum gives an approximate Fe<sup>2+</sup>/Fe<sup>3+</sup> ratio of 15/85=0.18. Moun and Rosenqvist<sup>16)</sup> postulated that bivalent iron Fe<sup>2+</sup> under fairly alkaline state oxidizes the oxygen in the pores resulting negative pressure in concrete and finally suck the aggressive solutions. If this is true, Fe<sup>2+</sup> that exist at areas between corroded and sound could play an important role in stimulating the ingress of sulfuric acid during corrosion process.

### (3) On-site Investigation of Concrete Structure

Results obtained by ICP analyses of core samples revealed the accumulation of high Fe-ions concentration (Table 3). At contact aeration of M treatment plant, Fe-ions was the highest at depth less than 8 mm from the surface. The carbonated depth was 4~8 mm and the surface pH was about 4~5. Concrete surface was covered with whitish soft powdered-like layer showing the gypsum or calcite and red staining spots scattering on the surface. The similar observation was also found at aeration tank of K treatment plant. High Fe-ions concentration was found at depth 7 mm and less. The concentration of H<sub>2</sub>S and CO<sub>2</sub> gasses were 0.9~14.8 ppm and 5500~6000 ppm, respectively. Severe corrosion occurred at anaerobic tank displayed by low surface pH of 1~3 and larger carbonation depth of 13 mm. Concentration of Fe-ions was higher in the middle and bottom core at depth approximately 8~13 mm where the existence of Fe-layer can be seen explicitly. At Y treatment plant, Fe-ions concentration was quite low compared to the previous plants. The H<sub>2</sub>S and CO<sub>2</sub> gasses concentration were not as high as the K treatment plant. This was probably due to frequent leaching of iron on the surface of concrete. Certain cores exhibited two zones with high Fe-ions as in second anaerobic tank of K treatment plant. The

**Table 3** On-site investigation of concrete structure.

Treatment plant	Compartment	Surface pH	Atmospheric H <sub>2</sub> S (ppm)	CO <sub>2</sub> (ppm)	Carbonation depth (mm)	Depth from surface (mm)	Fe-ion (%)
N	Contact Aeration (70m from top)	4-5	0.7	300	4-8	<8.0 12.0 16.0	4.11 2.73 1.58
	Contact Aeration (82m from top)	4-5	0.7	300	4-8	1.5- 4.5 4.5-12.0 15.0-16.0	0.86 2.92 1.14
K	Contact Aeration (40-80m from slab)	4-5	0.9	5500	4-5	Surface- 3.5 3.5- 7.0 7.0-9	2.05 1.95 1.91
	2nd Anaerobic Tank (5m above sewage line)	1-3	10.7	2400	13	Core Surface Middle Bottom	1.53 2.48 2.42
	3rd Anaerobic Tank (sewage line)	1	7.2	4000	12	<5.0 5.0-10.0 10.0-16.5	1.19 1.75 3.54
Y	2nd Anaerobic Tank (10m above sewage line)	2-4	3.1	1700	16	Surface- 5.0 5.0-19.0	0.92 0.86
	2nd Anaerobic Tank (sewage line)	2-4	3.1	1700	14	Surface- 3.5 3.5-10.0 10.0-16.5	1.03 0.82 0.89
	2nd Anaerobic Tank (10m below sewage line)	7			11	Surface- 6.0 6.0-12.0 12.0-18.0	0.83 0.96 0.67



**Fig.6** Fe-layer in concrete core of corroded plant.

**Table 4** Effect of carbonation on microbial corrosion.

Specimens	Carbonation rates (mm/year)	Fe-layer formation rate (mm/year)	Corrosion rates (mm/year)	Mass loss (%/year)
Control	4.3	3.67	2.1	11.0
0.5% CO <sub>2</sub>	16.4	5.42	3.8	13.9
5 % CO <sub>2</sub>	7.1	4.55	3.0	10.5

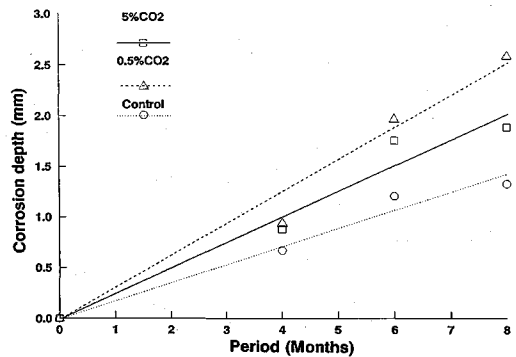
same finding was also detected by Tazaki *et al.*<sup>10)</sup>. The Fe-layer line was not straight due to different degree of sulfuric acid attack on concrete surface and the presence of aggregates and voids. The depth of Fe-line is more than the corrosion depth as shown in Fig.6.

**(4) Relationship between Corrosion Depth and Fe-layer Depth**

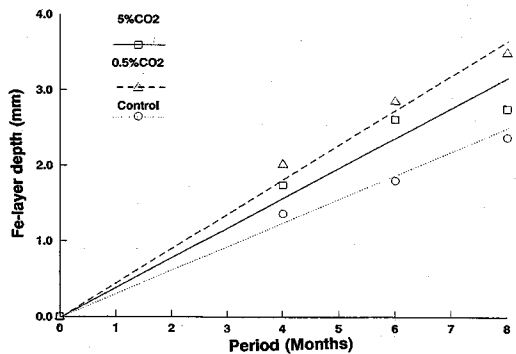
Relationship between Fe-layer depth and corrosion depth was investigated in the laboratory. Effect of carbonation on microbial corrosion of mortar specimens produced results as shown in Table 4. It shows that 0.5% specimens had the highest carbonation rate, corrosion rate as well as mass loss. It can be concluded that carbonation stimulated corrosion process. However, exposure to quite high CO<sub>2</sub> concentration slowed the corrosion process due to formation of much calcite.

When *T.thiooxidans* colonization and proliferation took place, surface pH of mortars dropped and sulfuric acid will gradually found the ways penetrating and attacking the sound layers. This eight months microbial corrosion experiment demonstrated that a drop of pH caused an increase of Fe-ions at Fe-layer (Table 2) and at the same time depth of Fe-layer from surface gradually increased.

Fig.7 shows that both corrosion and Fe-layer depths increased linearly with the increase in corrosion period. The corrosion rates for 5%, 0.5%, and control were 3.0, 3.8, and 2.1 mm/year, respectively. On the other hand, Fe-layer movement rates for 5%, 0.5%, and control were 4.55, 5.42, and 3.67 mm/year. These values are reasonable and higher than the corrosion rates taking consideration the fact that depth of Fe-layer was located deeper than the corrosion depth. Calculat-



(a) Corrosion depth



(b) Fe-layer depth

**Fig.7** Relationship between corrosion depth and Fe-layer depth.

ing the ratios between the corrosion depths and Fe-layer depths or corrosion rates and Fe-layer formation rates lead us to important information. Ratios for 5%, 0.5%, and control specimens were 1.52, 1.43, and 1.7, respectively. These values

indicate that the Fe-layer depth or Fe-layer formation rate were approximately 1.5 times higher or faster than the corrosion depth or corrosion rate.

#### 4. SUMMARY AND CONCLUSIONS

Findings about the behaviors of Fe-ions and Fe-layer could provide more valuable informations in our effort to effectively countermeasure the corrosion process. To rehabilitate and repair the corroded concrete effectively, the extent and severity of corrosion process should be examined and understood beforehand. Eventhough various factors such as H<sub>2</sub>S gas concentration, pH, turbulence flow, temperature, and *etc.* have been proposed<sup>17)</sup>, these methods are time and energy consuming. Rather simple and reliable indicators performed on-site are essential for sewer authorities or someone with minimal knowledge or background of microbial corrosion enable to determine the corrosion rate as well as the remaining service life of the structures.

Therefore, based on the results from laboratory and on-site investigations, Fe-layer can be an indicator to determine the microbial corrosion rate and remaining service life of concrete structures. On-site Fe-layer can be clearly visualized after removing the loosen corroded concrete materials with jet of tap water. Laboratory experiment indicated that Fe-layer depth from original surface was about 1.5 times larger than the corrosion depth. However, actual ratio value on site needs further investigation. Other factors such as the corrosion of steel bars of reinforced concretes should also be taken into consideration when analyzing this phenomena. Ratio of water-cement also influence the degree of discoloration since high water-cement ratio poses high iron content in the concrete as observed by Kong and Orbisson<sup>15)</sup>. Based on the results, the following conclusions are established :

1. Fe-ions migrated from inside of the specimen to the carbonated outer layer during carbonation process.

2. When microbial corrosion took place, Fe-ions accumulated and concentrated locally and eventually very thin Fe-layer was formed between the corroded and sound (carbonated) zone.

3. Formation of Fe-line behaved in similar manner with corrosion depth. The increase of corrosion depth was in agreement with the increase of Fe-layer depth.

4. Fe-layer divides concrete between corroded and sound in the case of microbial corrosion. Therefore, Fe-layer can be an indicator to measure the microbial corrosion rate of concretes.

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### コンクリートの微生物腐食を決定するための指標としての Fe 層の意義

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コンクリートの微生物腐食を表す指標として、腐食部分の先端に見られる Fe 層の存在理由とその意義を室内実験と現場調査により明示する。ICP での Fe イオンの分析から、炭酸化と微生物腐食による pH の低下によって、腐食先端に Fe イオンが集積することがわかった。この挙動は現場調査結果からも確かめられた。さらに腐食深さと Fe 層の位置には比例関係が見られた。微生物腐食速度の決定に Fe 層の追跡が有効と考えられる。