

Laboratory accelerated and outdoor durability testing of concrete with supplementary cementitious materials

Eisuke Nakamura* and Hiroshi Watanabe

(Received: March 1, 2015; Accepted: July 5, 2015; Published online: September 30, 2015)

Abstract: This paper aims to clarify the differences in durability test results of concrete containing supplementary cementitious materials (SCMs) for both laboratory-accelerated and outdoor exposure conditions. Twenty-six types of concrete specimens fabricated with and without SCMs were used for laboratory-accelerated and outdoor exposure tests to measure the resistance to carbonation and chloride ingress. An accelerated carbonation test and an immersion test were conducted in accordance with Japanese standard testing procedures, and outdoor exposure tests were performed at three test sites in Japan over 20 months. Although the testing conditions between these two approaches were substantially different, both results exhibited the same trend: the use of SCMs impaired the resistance to carbonation but improved that to chloride ingress. The carbonation depths estimated by the accelerated carbonation test were, however, slightly greater than those measured in the exposed specimens. This conservative estimation for the carbonation depth was assumed to be a result of overly severe testing conditions of the accelerated carbonation test. Additionally, differences in the chloride profiles of the specimens were found between those soaked in a NaCl solution and those exposed to outdoor conditions. These differences were prominent in the specimens containing high-volume SCMs because their chloride profiles were affected by carbonation observed at the concrete surface layer of the specimens. It should be emphasized that the chloride penetration resistance improved by the use of SCMs for the laboratory-accelerated condition was, on the other hand, relatively reduced for the outdoor exposure conditions where carbonation and chloride ingress progressed simultaneously in concrete.

Keywords: supplementary cementitious materials, carbonation, chloride ingress, laboratory-accelerated test, outdoor exposure test.

1. Introduction

Within the last several decades, a great deal of attention has been directed towards utilizing supplementary cementitious materials (SCMs) for reducing carbon dioxide emissions in the concrete construction industry. The replacement of Portland cement with SCMs is one of the reasonable options to reduce carbon dioxide emissions. It is, however, widely accepted that the use of high-volume SCMs affects the properties of fresh and hardened concrete. In the interest of environmental responsibility, durability, in terms of sustainable use of concrete structures, is another key property. The durability of concrete containing high-volume SCMs should be evaluated using reliable testing procedures.

Laboratory-accelerated and outdoor exposure tests are employed to determine the resistance of concrete to carbonation and chloride ingress in artificial and actual environmental conditions, respectively. The laboratory-accelerated tests allow for quick results but adopt substantially different testing procedures from those of natural exposure conditions. On the other hand, outdoor exposure tests evaluate the durability of concrete under actual environmental conditions but require long test periods. There are tremendous amounts of researches on the durability of concrete containing SCMs, and investigations of outdoor-exposed specimens and existing concrete structures have been performed around the world [1, 2, 3, 4, 5]. The differences in the durability test results derived from those two approaches are, however, not fully clear for concrete containing high-volume SCMs. Additionally, little is known about whether conventional laboratory-accelerated tests are applicable in evaluating the durability of concrete fabricated with high-volume

Corresponding author Eisuke Nakamura is a Senior Researcher at the Public Works Research Institute, Japan
Hiroshi Watanabe is Director of the Materials and Resources Research Group at the Public Works Research Institute, Japan

Table 1 – Concrete mixtures and fresh concrete properties

Mixture No.	W/B (%)	B (kg/m ³)	B = OPC+HPC+BS4+BS6+FA Binder composition (%)					Slump (mm)	Air (%)
			OPC	HPC	BS4	BS6	FA		
H40	40	413	--	100	--	--	--	115	4.6
H40B430			--	70	30	--	--	105	3.8
H40B450			--	50	50	--	--	135	5.1
H40B630			--	70	--	30	--	135	4.7
H40B650			--	50	--	50	--	145	4.8
H40B670			--	30	--	70	--	130	4.9
H40F10			--	90	--	--	10	95	4.2
H40F20			--	80	--	--	20	140	4.5
H40F30			--	70	--	--	30	120	4.3
H40S			--	100	--	--	--	120	4.6
H40B430S			--	70	30	--	--	100	3.9
H40B650S			--	50	--	50	--	125	4.7
H40F20S			--	80	--	--	20	105	3.1
N35	35	471	100	--	--	--	--	145	4.7
N35B50			50	--	50	--	--	145	4.3
N35B85			15	--	85	--	--	125	5.1
N35F20			80	--	--	--	20	120	3.5
N35F40			60	--	--	--	40	145	4.5
N50	50	330	100	--	--	--	--	140	5.2
N50B50			50	--	50	--	--	135	4.3
N50B70			30	--	70	--	--	125	4.5
N50B85			15	--	85	--	--	115	4.0
N50F20			80	--	--	--	20	115	4.4
N50F30			70	--	--	--	30	110	4.6
N50F40			60	--	--	--	40	145	4.0
N50B50F20			30	--	50	--	20	120	4.6

- Note:
- 1) Water content = 165 kg/m³, Coarse aggregate content = 968 kg/m³;
 - 2) W/B = water-to-binder ratio, W = tap water, B = cementitious binder;
 - 3) OPC = Ordinary Portland cement (3.16 g/cm³, 3,300 cm²/g), HPC = High early strength Portland cement (3.14 g/cm³, 4,490 cm²/g), BS4 = Ground granulated blast-furnace slag (2.89 g/cm³, 4,400 cm²/g), BS6 = Ground granulated blast-furnace slag (2.91 g/cm³, 5,950 cm²/g), FA = Fly ash (2.30 g/cm³, 4,280 cm²/g);
 - 4) Fine aggregate = natural sand (2.56 g/cm³), Coarse aggregate = crushed gravel (2.67 g/cm³);
 - 5) The specimens adopting four concrete mixtures with 'S' in their Mixture No. were steam cured under the following conditions: 2 h from 20–50°C, 6 h at 50°C (Max. Temperature), and 5 h from 50–20°C. The specimens employing other 22 concrete mixtures were cured in water at 20°C; and
 - 6) The measurement of slump and air content was conducted in accordance with JIS A 1101 and JIS A 28, respectively.

SCMs especially in the environmental conditions where carbonation and chloride ingress progress simultaneously.

The purpose of this paper is to clarify the differences in the durability test results derived from laboratory-accelerated and outdoor exposure tests by using concrete specimens fabricated with and without SCMs, such as ground granulated blast-furnace slag (GGBF slag) and fly ash. Twenty-six types of concrete specimens were prepared at the Public Works Research Institute, and subsequently used for the laboratory-accelerated and outdoor exposure tests. Two laboratory-accelerated tests, an accelerated carbonation test and an immersion test, were conducted in accordance with Japanese standard testing procedures. The 20-month outdoor ex-

posure test at three test sites in Japan was performed to investigate the resistance of concrete to carbonation and chloride ingress under actual environmental conditions. The results derived from these two approaches were compared in order to study the applicability of conventional laboratory-accelerated tests of concrete containing high-volume SCMs.

2. Experimental program

2.1 Concrete specimens

Concrete specimens using 26 types of concrete mixtures with and without SCMs, such as GGBF slag and fly ash, were prepared for the laboratory-accelerated and outdoor exposure tests. The con-

crete mixtures and fresh concrete properties of the specimens are presented in Table 1. All materials conformed to Japanese Industrial Standards (JIS), and were readily available in the Japanese market. The concrete mixtures using high early strength Portland cement adopted relatively low replacement levels of SCMs and water-to-binder ratios because these mixtures were deemed to be employed for prestressed concrete structures that usually required the rapid strength development at early ages. On the other hand, those using ordinary Portland cement enabled to set relatively large replacement levels of SCMs for the application to plain or reinforced concrete structures.

The specimens were fabricated in a laboratory at a temperature of 20°C and a humidity of 60%. A forced-action biaxial mixer was used to mix the materials in the following two steps: 30-second mixing of cementitious materials and aggregates, and 90-second mixing after the addition of water and chemical admixtures. The target values of slump and air content were 12 ± 2.5 cm and $4.5 \pm 1.5\%$, respectively. The specimens adopting 22 concrete mixtures were cured in water at a temperature of 20°C for 28 days from the day after casting. On the other hand, the specimens employing four concrete mixtures, H40S, H40B430S, H40B650S, and H40F20S, were cured under procedures using high temperature steam for 13 hours, and then placed in water at a temperature of 20°C for 28 days. After these curing stages were complete, the specimens were brought to the laboratory - accelerated and outdoor exposure tests.

2.2 Laboratory-accelerated test

An accelerated carbonation test was conducted in accordance with the JIS A 1153 standard [6] in order to measure the resistance to carbonation in artificial testing conditions. One prism ($100 \times 100 \times 400$ mm) was fabricated for each concrete mixture. Two side surfaces (100×400 mm surfaces facing each other) were to be exposed while the other four surfaces were sealed with epoxy resin to avoid carbonation progression. The accelerated carbonation test was started at 56 days from the day after casting. The prisms were stored in a controlled storage chamber at the following conditions: a carbon dioxide concentration of $5 \pm 0.2\%$, a temperature of $20 \pm 2^\circ\text{C}$, and a humidity of $60 \pm 5\%$. An approximately 60-mm-thick slice was broken off from each prism at 1, 4, 8, 13, and 26 weeks after the start of the test, and the carbonation depths were measured at five points on each exposed surface by spraying a phenolphthalein solution. Based on the measured carbonation depths, the carbonation rate was determined using the following equation:

$$y = \alpha\sqrt{t} \quad (1)$$

where

y = measured carbonation depth (mm),

α = carbonation rate (mm/ $\sqrt{\text{week}}$), and

t = measurement time of carbonation depth (week).

Meanwhile, an immersion test based on the JSCE-G 572 standard [7] was performed to determine the resistance to chloride ingress in artificial testing conditions. Cylinders ($\phi 100 \times 200$ mm) were prepared of each concrete mixture, and shaped into 150-mm-thick cylinders by removing 25-mm-thick disks from both ends after the age of 28 days. The bottom and side surfaces were sealed with epoxy resin to prevent the penetration of chloride, and subsequently soaked in a 10% NaCl solution. The cylinders were sliced into 10-mm-thick disks from the top surface at 20 months after the start of the test. This test duration was equivalent to that of the outdoor exposure test. The chloride content of each disk was measured in accordance with the JIS A 1154 standard [8] to obtain the chloride profile. The chloride diffusion coefficient was determined by using the measured chloride profiles and the following equation:

$$C(x, t) - C_i = (C_0 - C_i) \left\{ 1 - \operatorname{erf} \frac{x}{2\sqrt{D_{ap}t}} \right\} \quad (2)$$

where

$C(x, t)$ = chloride content (kg/m^3) measured at depth x (m) and time t (s),

C_i = initial chloride content (kg/m^3),

C_o = surface chloride content (kg/m^3),

x = depth (m),

D_{ap} = chloride diffusion coefficient ($\times 10^{-12}\text{m}^2/\text{s}$),

t = test duration (s), and

erf = error function.

2.3 Outdoor exposure test

The setup and climate conditions at the three test sites in Japan are presented in Fig. 1. Average air temperature, average ambient humidity, and total amount of precipitation were calculated from data provided by the Japan Meteorological Agency [9]. One of the test sites is on the grounds of the Public Works Research Institute in Tsukuba, Japan. Because this test site is located inland where no de-icing salts are used, it is reasonable to assume that chloride does not come into contact with the specimens. On the contrary, the other two test sites are located along the coastlines at Niigata and Okinawa, Japan. This implies that a significant amount of airborne salt reaches the specimens at these two test sites. The Niigata site is in a cold region while the

	Tsukuba	Niigata	Okinawa
Average temperature (°C)	15.5	14.8	23.3
Average humidity (%)	72.9	77.3	75.5
Total precipitation (mm)	2,580	4,830	4,085

Fig. 1 – Setup and climate conditions of the three test sites in Japan

Okinawa site is in a subtropical region; the ambient air temperature is substantially different between the two test sites as well.

A detailed schematic of the specimen ($100 \times 100 \times 200$ mm) prepared for the outdoor exposure test is illustrated in Fig. 2. Five surfaces, excluding the exposed side surface (100×200 mm), were sealed with a durable coating material to prevent carbonation and chloride ingress. The specimens were collected 20 months after the start of the test, and subsequently used to measure the carbonation depth and the chloride profile as with the laboratory-accelerated test; the carbonation depth was measured by spraying a phenolphthalein solution on the split surface, and the chloride profile was obtained by measuring the chloride content in accordance with the JIS A 1154 standard.

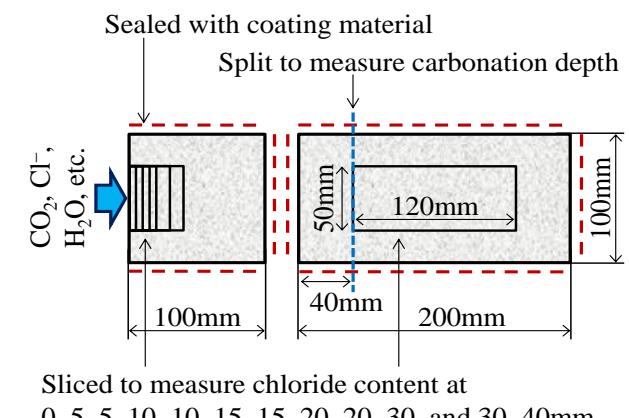


Fig. 2 – Detailed schematic of exposed specimen

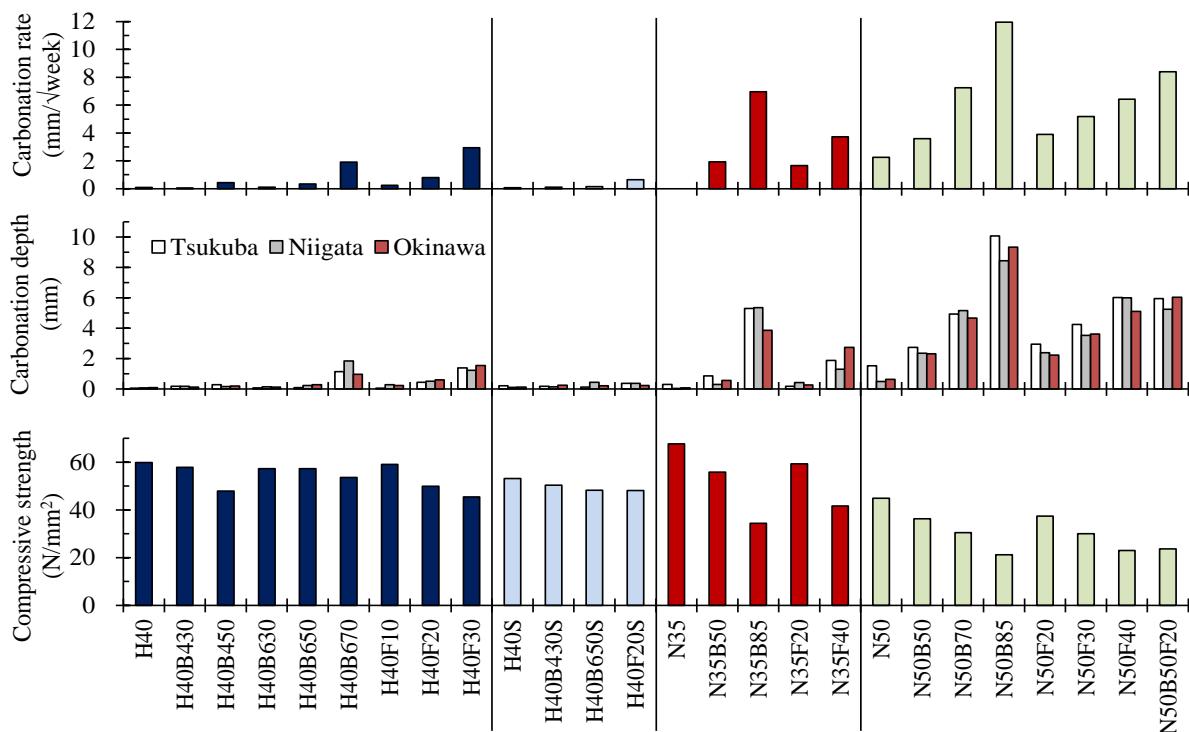


Fig. 3 – Carbonation rate, carbonation depth, and compressive strength

3. Results and discussions

3.1 Resistance to carbonation

The carbonation rate, carbonation depth, and compressive strength of all specimens using 26 concrete mixtures are shown in Fig. 3. The carbonation rate was derived from the JIS A 1153 accelerated carbonation test, and the carbonation depth was measured using the specimens exposed at the three test sites for 20 months. The carbonation rate will be used to compute the estimated carbonation depth by calibrating the differences in the carbon dioxide concentration between the accelerated carbonation test and the outdoor exposure test later. The compressive strength was the average of three cylinders ($\phi 100 \times 200$ mm) at the age of 28 days. For specimens of the same cement type and water-to-binder ratio, those containing larger amounts of SCMs showed an increase in the carbonation ratio and the carbonation depth and a decrease in the compressive strength. An increase in the carbonation depth was found for each test site. The differences in the carbonation depths among the three test sites were not significant. These findings suggest that the carbonation depths of the exposed specimens depend strongly on the differences in the binder compositions but not on outdoor exposure conditions at the three test sites.

The relationship between the carbonation depth and compressive strength of the exposed specimens at the age of 28 days is presented in Fig. 4. The carbonation depth decreased as the compressive strength increased. In other words, the carbonation resistance improved with the increased compressive strength. This relationship was assumed to indicate that the increased compressive strength indirectly exhibit relatively dense pore structures inhibiting the penetration of carbon dioxide into concrete. Special attention, however, should be paid to the specimens containing high-volume SCMs, such as H40B670, N35B85, N50B70, and N50B85,

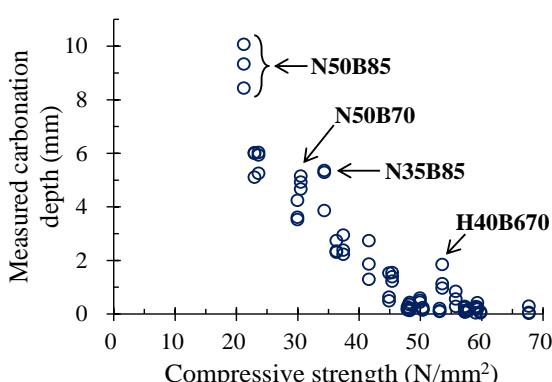


Fig. 4 – Relationship between measured carbonation depth and compressive strength

because these specimens have larger carbonation depths than those fabricated with smaller amounts of SCMs at a given compressive strength. On this basis, it is not recommended to use the compressive strength as an indirect index to predict the carbonation resistance of concrete containing high-volume SCMs.

The carbonation depths measured for the exposed specimens are compared in Fig. 5 to those estimated using the carbonation rate derived from the accelerated carbonation test. In this figure, the estimated carbonation depth was calculated using the following equation:

$$C = A_{ACT} \cdot \sqrt{\frac{CO_2}{CO_{2ACT}}} \cdot \sqrt{t} \quad (3)$$

where

C = estimated carbonation depth (mm),

A_{ACT} = carbonation ratio given by the accelerated carbonation test (mm/ $\sqrt{\text{week}}$),

CO_2 = average carbon dioxide concentration in the atmosphere (0.03931% [9]),

CO_{2ACT} = carbon dioxide concentration in the accelerated carbonation test (5%), and

t = concrete age (weeks).

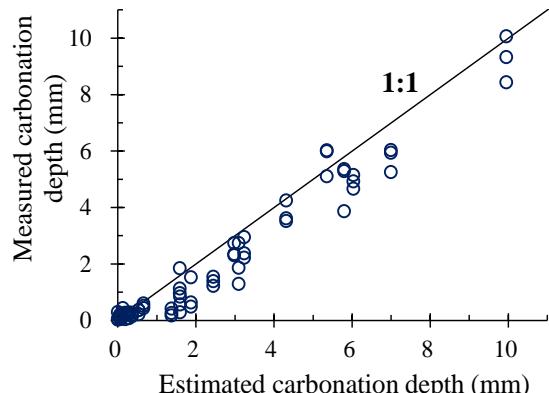


Fig. 5 – Relationship between measured and estimated carbonation depths

The estimated carbonation depths were in agreement with the measured results; the accelerated carbonation test results correlated better with the measured carbonation depths than with the compressive strengths. Additionally, most of the estimated values were slightly greater than the measurement results. These facts indicate that the accelerated carbonation test was useful for quickly obtaining the conservative estimations for the specimens containing high-volume SCMs. These conservative estimations were assumed to be induced by severe testing conditions employed in the accelerated carbonation test, including dry condition and

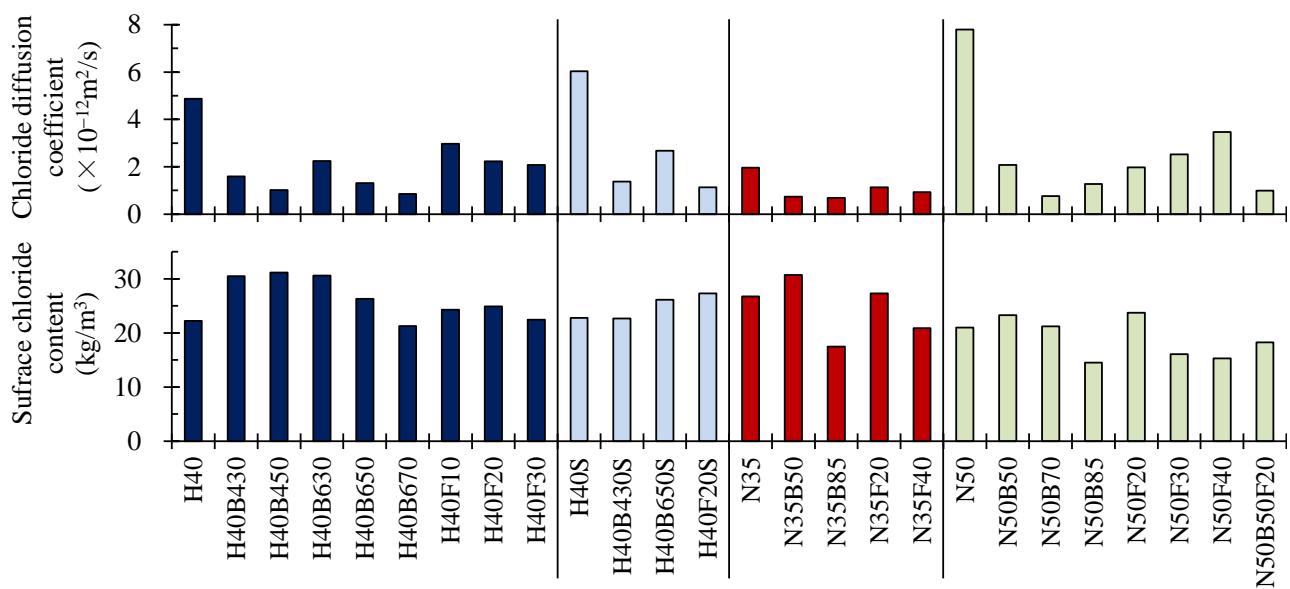


Fig. 6 – Chloride diffusion coefficient and surface chloride content

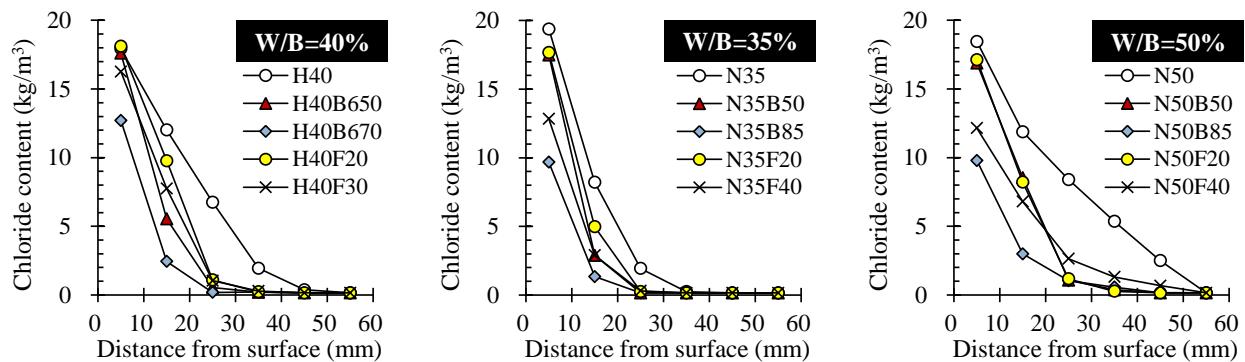


Fig. 7 – Chloride profiles of specimens immersed in NaCl solution

high carbon dioxide concentration. It is important to note that the carbon dioxide concentration used in the JIS A 1153 standard is 5%, a value much greater than those adopted in other test standards [10, 11] and those measured in the atmosphere [9]. Moreover, while the specimens exposed to outdoor conditions were subjected to moisture exposure due to rainfall and high humidity, those used for the accelerated carbonation test were stored in a chamber with constant humidity. It should be noted that these severe testing conditions of the accelerated carbonation test are viewed as unavoidable when quick results by using laboratory-accelerated techniques are desired.

3.2 Resistance to chloride ingress

The chloride diffusion coefficient and surface chloride content derived from the immersion test are illustrated in Fig. 6. These values were computed using the chloride profiles of the specimens soaked in a NaCl solution for 20 months. Typical chloride profiles of the specimens with respective

water-to-binder ratios are shown in Fig. 7. It is clear that the specimens made with SCMs yield lower chloride diffusion coefficients than those without SCMs. The decrease in the chloride diffusion coefficients due to the use of SCMs was greater than that obtained by the reduction of the water-to-cement ratios of the specimens made only with Portland cement. In other words, the use of the SCMs was found more effective in improving the resistance of concrete to chloride ingress than did the adjustment of the water-to-cement ratio. On the other hand, the surface chloride content was greater for the specimens containing GGBF slag, which was assumed to contribute to the development of dense pore structures and to the binding of chloride in concrete. It is, however, interesting to note that the specimens with a GGBF slag replacement ratio of 85%, N35B85 and N50B85, and those with a fly ash replacement ratio of 40%, N35F40 and N50F40, showed low surface chloride contents. This tendency could be a characteristic phenomenon of the specimens containing high-volume SCMs.

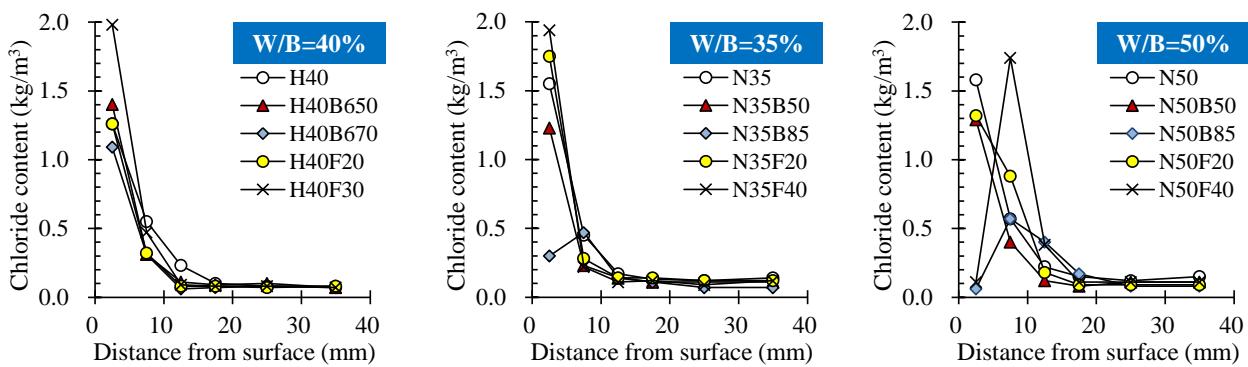


Fig. 8 – Chloride profiles of specimens exposed at Niigata

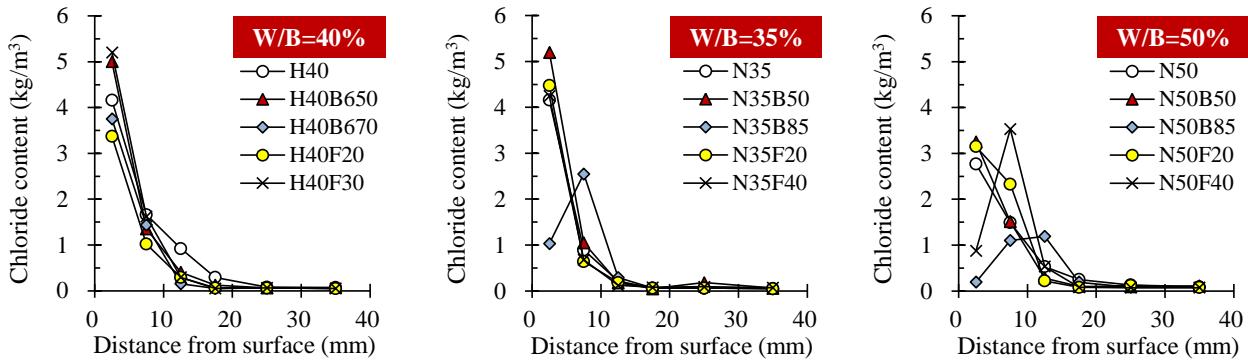


Fig. 9 – Chloride profiles of specimens exposed at Okinawa

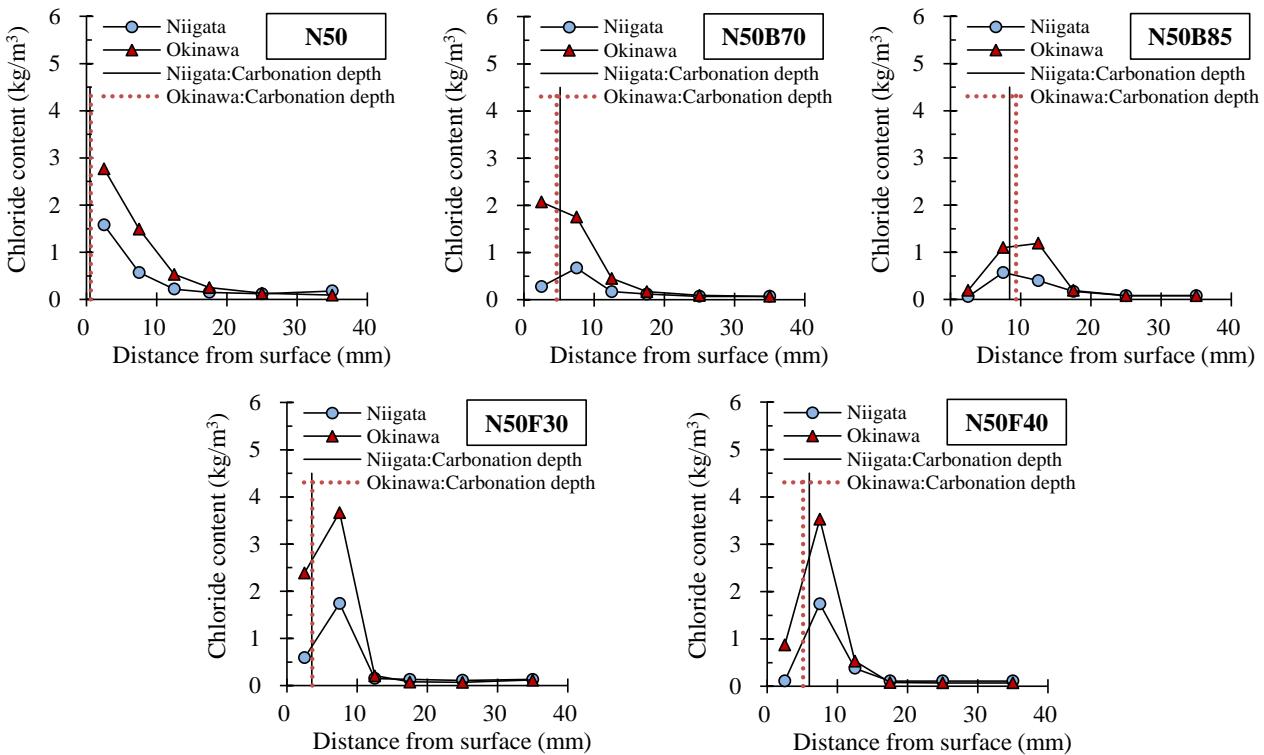


Fig. 10 – Relationship between carbonation depth and chloride profile

Typical chloride profiles of the specimens exposed at the Niigata and Okinawa sites for 20 months are presented in Fig. 8 and Fig. 9, respectively. The chloride contents of the exposed specimens are lower than those of the immersed specimens. This difference is attributed to the variance in

the testing conditions between the immersion test and the outdoor exposure test; the immersed specimens were soaked in a 10% NaCl solution whereas the exposed specimens were subjected to airborne salt from the coast line. Moreover, the chloride contents measured in the Okina was specimens were

larger than those observed in the Niigata specimens because a large amount of airborne salt was assumed to have reached the Okinawa specimens, which were set up adjoining a tidal zone. The chloride profiles of the exposed specimens with a water-to-binder ratio of 40% indicated that the use of SCMs was effective at enhancing the resistance to chloride ingress. As mentioned previously, this trend was also observed in the results obtained for the immersed specimens. On the other hand, for the exposed specimens with water-to-binder ratios of 35% and 50%, the specimens containing high-volume SCMs, such as N35B85, N50B70, and N50B85, showed that the chloride content at the outermost layer was lower than that inside the specimen. This inverse distribution of the chloride content was not observed in the specimens soaked in a 10% NaCl solution.

In order to clarify the reason for the difference in the chloride profiles between the immersed and exposure specimens, typical relationships between the carbonation depth and the chloride profile of the exposed specimens are presented in Fig. 10. With regards to the carbonation depth and the chloride profile of the specimens, with the exception of N50, it is notable that the chloride ingress was induced by the progression of carbonation at the concrete surface layer of the specimens. As a result, the improved chloride resistance achieved by the use of SCMs was relatively reduced for the outdoor exposure conditions where carbonation and chloride ingress progressed simultaneously in concrete. Therefore, it should be emphasized that the immersion test is not able to examine the effect of carbonation at the surface concrete layer of the specimens and that this effect must be considered in the evaluation of the chloride resistance of concrete containing high-volume SCMs. The simultaneous development of carbonation and chloride ingress also made it difficult to compute the chloride diffusion coefficients of the exposed specimens.

4. Conclusions

The conclusions derived from this experimental investigation are summarized as follows:

- (1) Although the testing conditions between the laboratory-accelerated and outdoor exposure tests were substantially different, both results exhibited the same trend; the use of SCMs impaired the resistance to carbonation but improved that to chloride ingress.
- (2) The carbonation depths obtained in the outdoor exposure test were proportional to those estimated using the accelerated carbonation test results even for the specimens containing high-

volume SCMs. Most of the estimated carbonation depths were conservative because the accelerated carbonation test employed overly severe testing conditions to more quickly evaluate the carbonation resistance.

- (3) The use of SCMs was found more effective in improving chloride resistance than did the reduction of the water-to-cement ratio. The chloride profiles of the exposed specimens containing high-volume SCMs were, however, affected by carbonation at the concrete surface layer of the specimens. This phenomenon indicated that the chloride resistance improved by the use of SCMs was relatively reduced for the outdoor exposure conditions where chloride ingress and carbonation progressed simultaneously in concrete.

The outdoor exposure test presented in this paper is planned to be implemented for 20 years. The progress of the continued research work will be reported without interruption. Additionally, it should be noted that this research program has been started focusing only on a Japanese context; that is to say, concrete materials, laboratory-accelerated test procedures, and outdoor exposure conditions available in Japan. Further research programs incorporating international perspectives are indispensable for achieving the sustainable use of concrete containing high-volume SCMs.

Acknowledgements

The authors are sincerely grateful to the project members of the Collaborative Research Project on Effective Use of Low-carbon Cements for their contributions. Opinions, findings, conclusions, and recommendations in this paper are those of the authors.

References

1. Stark, D.C.; Kosmatka, S. H.; Farny, J.A.; and Tennis, P.D. (2002) "Performance of concrete specimens in the PCA outdoor test facility," RD124, Portland Cement Association, 36 pp.
2. Tamg, L. (2005) "CHLORTEST Resistance of concrete to chloride ingress – From laboratory tests to in-field performance," WP3 Report – Collection of in-field data, 29 pp.
3. Thomas, M.D.A.; Bremner, T.; and Scott, A.C.N. (2011) "Actual and modeled performance in a tidal zone," Concrete International, 11, pp. 23-28.
4. Tang, L. (2013) Resistance of Concrete to Chloride Ingress, Spon Press, 241 pp.
5. Gjørv, E.O. (2014) Durability Design on Concrete Structures in Severe Environments, Second Edition, CRC Press, 254 pp.
6. Japanese Industrial Standards Committee (2012) JIS A 1153:2012, Method of accelerated carbonation test for concrete.
7. Japan Society of Civil Engineers (2013) JSCE-G

- 572-2013, Test method for apparent diffusion coefficient of chloride ion in concrete by submergence in salt water.
- 8. Japanese Industrial Standards Committee (2012) JIS A 1154: 2012, Methods of test for chloride ion content in hardened concrete.
 - 9. Japan Meteorological Agency, <http://www.jma.go.jp>. (Dec. 20th 2013)
 - 10. CEN (2007) DD CEN/TS 12390–10:2007, Testing hardened concrete – Part 10: Determination of the relative carbonation resistance of concrete.
 - 11. CEN (2004) BS EN 13295:2004, Products and systems for the protection and repair of concrete structures – Test methods – Determination of resistance to carbonation.