Scientific paper

Study on the Method of Measuring the Chloride Threshold Value of Corrosion and Estimation of the Values in Durability Design of Concrete Structures

Kenichi Horiguchi1*, Toshinobu Yamaguchi2, Tsuyoshi Maruya3 and Koji Takewaka2

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Abstract

In this study, a method was developed to estimate accurately the chloride threshold value for corrosion initiation in reinforced concrete. In this method, chlorides are supplied through a part of the specimen surface, and half-cell potential between the rebar and reference electrode embedded in a concrete specimen was measured in every ten minutes. A sudden drop of half-cell potential was observed as the continuation of the measurement. At that time, the specimen was broken and corrosion of rebar on its narrow surface was confirmed. The chloride threshold value can be estimated by determining the chloride concentration at the time of the potential drop. The estimated chloride threshold values for normal Portland cement concrete with cement content between 254 and 446 kg/m³ are in the range of 1.6 to 3.6 kg/m³. This paper is an English translation from the authors’ previous work [Horiguchi, K., Yamaguchi, T., Maruya, T. and Takewaka, K., (2015). “A study on the method of measuring the chloride threshold value of corrosion and on the estimation of the values." Journal of JSCE, Ser. E2, 71(2), 107-123. (in Japanese)].

1. Introduction

In the Standard Specifications for Concrete Structures of the JSCE (Japan Society of Civil Engineers) revised in 2007 (JSCE 2007), verification for durability on chloride attack of reinforced concrete is based on the condition that corrosion should not be generated in steel during the service period, while also confirming that the total chloride concentration at the steel surface position should be below the chloride threshold value for corrosion initiation (hereinafter, the chloride threshold value). Furthermore, this chloride threshold value should be determined by referring to the actual measured results of similar structures or experiment results, but if not based on these results, 1.2 kg/m³ may be used. This value has been adopted as the value on the safety side, because the result of the exposure experiment in the real environment was about 1.2 to 2.4 kg/m³ (JSCE 2007). As described above, the chloride threshold value 1.2 kg/m³ was uniformly used regardless of the mix proportion of concrete, so excessive cover was sometimes required for reinforced concrete structures placed in severe salt damage environments such as splash zones. Therefore, in the Standard Specifications for Concrete Structures revised in 2012 (JSCE 2012), the chloride threshold values for each type of binder and each type of water/binder ratio were specified, reflecting a part of the results of a previous study by the authors (Horiguchi et al. 2015). The same evaluation method has been inherited in the version revised in 2017 (JSCE 2017).

Regarding the chloride threshold value, although there is much knowledge based on the results of actual structural measurements and experimental results of specimens obtained in Japan and overseas, the current situation is that a unified view has not been obtained (JSCE 2009). This is because it is difficult to quantify the chloride threshold value with high accuracy by conventional research methods. The reasons why highly accurate quantification is difficult are as follows: (1) the relation between the experimental conditions simulating the corrosion of steel in concrete and the corrosion generation conditions of steel in actual concrete is not clear, (2) it is difficult to clearly ascertain the point in time when corrosion occurs on the steel in concrete, and to accurately measure the total chloride ion quantity on the steel surface at that point, and (3) there are variations in the measurement results even under the same test conditions and measurement method.

For these reasons, the purpose of this study is to establish a measurement method that is capable of quantitatively evaluating the chloride threshold value of steel embedded in reinforced concrete, and to obtain a quantitative threshold value.

2. Previous studies on the chloride threshold value for corrosion initiation

A number of experiments have been carried out in Japan and overseas for many years, regarding the chloride
threshold value (Alonso et al. 2000; Bamforth 1999; Brian et al. 1987; David 2009; David et al. 2004; Dehwah 2003; Hartt et al. 2008; Mohammed et al. 2001; Ormellese et al. 2007; Radhakrishna et al. 2005; Ryou et al. 2008; Vidal et al. 2007). However, since there are a wide variety of experimental methods and conditions, the results obtained are also different. As factors affecting the experimental results, the following are mentioned: (1) contact method between steel and chloride ion, (2) supply method of chloride ion, (3) materials and mix proportions of the concrete used, and (4) confirmation method concerning corrosion occurrence.

As contact methods of steel and chloride ion, there are methods such as that of embedding steel material in a cement hardened body like mortar or concrete and immersing it in salt water (Yonezawa et al. 1986, 1987; Oh et al. 2003, 2004), and a method of immersing steel material in an aqueous solution which reproduced the pore solution in the cement hardened body (Hausmann 1967; Diamond 1986; Ishida et al. 2007a).

As methods of supplying chloride ion to a cement hardened body with embedded steel, there are several. One is method of mixing as initial salinity when it is kneaded (Oh et al. 2003, 2004; Otsuki et al. 1985), and another is a method of soaking in salt water (Yonezawa et al. 1986, 1988; Oh et al. 2003, 2004), and a method of immersing steel material in an aqueous solution which reproduced the pore solution in the cement hardened body (Hausmann 1967; Diamond 1986; Ishida et al. 2007a).

As described above, there are many factors that affect the chloride threshold value and measurement accuracy. Therefore, there are large differences among the measurement results of past experiments.

Incidentally, the chlorine existing in concrete is classified into immobilized chlorine and chloride ion according to its form of existence shown in Fig. 1, and further, immobilized chlorine is classified into solid phase chlorine and adsorbed chlorine (Maruya et al. 1998). Expressing the threshold value of total chloride concentration when corrosion occurs in steel involves; (1) expressing the total chloride ion mass for per unit volume of concrete as a concentration (kg/m³) (hereinafter, notation for total amount of concrete), (2) expressing the total chloride ion mass for the mass of a binder as a mass percent (mass % of binder) (hereinafter, notation for mass % of binder), and (3) expressing as the molar concentration ratio of the chloride ion concentra-

![Fig. 1 Form of existence of chlorine in concrete.](image)

| Literature | Experimental conditions | Molar concentration ratio [Cl⁻]/[OH⁻] | Total amount of concrete (kg/m³) | Mass % of binder |
|------------|-------------------------|--------------------------------------|---------------------------------|-----------------|
| Hausmann (1967) | In solution | pH 11.6 - 13.2 | 0.50 - 0.83 | 0.46 - 3.45 | 0.15 - 1.15 |
| Gouda (1970) | In solution | pH 11.8 - 13.3 | 0.27 - 0.57 | 0.43 - 3.21 | 0.14 - 1.07 |
| Diamond (1986) | In solution | pH 12.6 | 1.10 - 5.40 | 2.03 - 4.36 | 0.68 - 1.45 |
| Li et al. (2002) | In solution | pH 13.6 | 0.50 - 5.00 | 4.20 - 12.7 | 1.40 - 4.23 |
| Ishida et al. (2007a) | In solution | pH 12.5 | 0.25 - 0.30 | 0.89 - 0.98 | 0.30 - 0.33 |
| Yonezawa et al. (1986, 1987) | In solution | pH 13.0 | 0.10 - 0.20 | 1.00 - 1.39 | 0.33 - 0.46 |
| Hussain et al. (1995, 1996) | In hardened body | pH 12.95 - 13.20 | 1.28 - 2.00 | 3.22 - 5.25 | 1.07 - 1.75 |
| Oh et al. (2003, 2004) | In hardened body | - | 0.16 | - | 0.27 |
| Hurley et al. (1986, 1987) | In hardened body | - | 0.25 - 0.34 | - | - |
| Page et al. (1985) | In hardened body | Silica fume 30% | 62 | - | - |
| Kitago et al. (2000) | In hardened body | pH 12.0 - 13.0 | 1.00 - 3.00 | 1.00 - 1.69 | 0.33 - 0.56 |
Table 2 Factors for an experiment on the quantitative evaluation method.

| Factor                          | Method of supplying chloride ion | Dry and wet condition | Maximum dimension of coarse aggregate | Number of rebar | Measurement method of half-cell potential | Measurement frequency of half-cell potential |
|--------------------------------|----------------------------------|-----------------------|--------------------------------------|-----------------|------------------------------------------|---------------------------------------------|
| Whole immersion experiment     | Immerse the whole specimen in salt water | Wet and dry Immersion 3 days, Dry 4 days | Cover 15 mm $g_{\text{max}}$ 13 mm | 1 | Manual measurement by the reference electrode from the surface of specimen | Once a week |
| Partial contact experiment     | Contact salt water to specimen surface | Always wet a part of the surface | Cover 20 mm or 20, 25 mm $g_{\text{max}}$ 13 mm | 1 or 2 | Continuous automatic measurement with buried reference electrode | Once in 10 minutes |

As described above, a notation for the total amount of concrete and a notation for mass % of the binder are based on the total amount of chloride ion. On the other hand, a notation for molar concentration ratio focuses on chloride ions only, which are dissolved in the pore solution and can be moved freely, while the immobilized chlorine is excluded. It is reasonable to express the chloride threshold value by a notation for the molar concentration ratio, because theoretically the chloride, which affects the corrosion of steel in concrete, is chloride ion in the pore solution, and the generation of corrosion is considered to depend on the amount of chloride ion and hydroxide ion. However, it is not practical to check salt damage at the molar concentration ratio. It is also not practical to check at mass % of the binder. It is considered that the molar concentration ratio depends on the type of cement and the amount of binder, but it is not practical to check the molar concentration ratio because it is difficult to determine the details at the stage of designing the concrete structure. Therefore, in this paper, the chloride threshold value is based on a notation for the total amount of concrete (kg/m$^3$), the same as the specification in JSCE.

3. Experiment to determine a quantitative evaluation method for the chloride threshold value for corrosion initiation

3.1 Outline of the experiment
In this study, a reinforced concrete specimen was fabricated, and chloride ion was supplied from the outside to corrode the steel inside, and the chloride threshold value was determined from the total chloride concentration distribution in the concrete at the time of corrosion. The is intended to measure the chloride threshold value as directly as possible by approaching the exposure of the specimen close to the condition of a reinforced concrete structure placed in a salt damage environment. Therefore, it is important to ascertain the corrosion generation point of steel materials accurately. First, an experiment to determine the quantitative evaluation method for the chloride threshold value was conducted. In this experiment, the measurements of the half-cell potential and visual observation of the existence of corrosion by disassembling the specimen were carried out.

Table 2 shows the experimental factors for the quantitative evaluation method. The experiment on the quantitative evaluation method was carried out by two methods; the method of repeated drying and wetting by immersing the whole specimen in salt water (hereinafter, the whole immersion experiment), and the method involving constant contact with a part of the specimen.
surface in salt water (hereinafter, the partial contact experiment). In addition to the method of supplying chloride ion, the following were also used as experimental factors; dry and wet conditions, cover, number of reinforcing bars to be buried in the specimen, and measuring method and measuring frequency of half-cell potential.

3.2 Specimen

Figures 2 and 3 show the shape and size of the specimen and the arrangement of reinforcing bars in the whole immersion experiment, and Figs. 4 to 7 show the shape and dimensions of the specimen in the partial contact experiment and the arrangement of the reinforcing bar and the lead reference electrode. The shape of the test specimen was a rectangular prismatic body, 100 × 100 × 400 mm in both experiments. In the whole immersion experiment, a single rebar specimen was prepared by burying one bar of SD 295A, D19 to a depth of 15 mm, while in the partial contact experiment, a single rebar specimen and a double rebar specimen were prepared. In the single rebar specimen, one bar of SD 295A, D19 was buried to a depth of 20 mm. In the double rebar specimen, two bars of SD 295A, D19 were buried to a depth of 20 mm and 25 mm. Furthermore, in the specimen for the partial contact experiment, a lead reference electrode with a diameter of 10 mm was placed as shown in Figs. 4 to 7. The reason why the double rebar specimen was manufactured is that even if it is not possible to ascertain clearly when corrosion has occurred in the rebar, if the small cover rebar is corroded and the large cover rebar is not, at least it is possible to know the chloride ion concentration that does not cause corrosion.

Tables 3 and 4 show the mix proportions and materials used for the experiment specimen in the quantitative evaluation method. The specimens for the whole immersion experiment were made by ordinary cement concrete in W/B 35% and blast furnace cement concrete in W/B 45%. The blast furnace slag fine powder used was a mixture of gypsum. The proportion of gypsum was 2.0% in SO3 conversion. Four specimens were manufactured for each mix proportion. On the other hand, in the partial contact experiment, using ordinary cement as a binder and concrete with W/B 65%, two bodies of a single bar specimen and two bodies of a double rebar specimen similar to the above; four bodies in total were fabricated.

The compressive strength of each concrete after 28 days of standard curing was 67.9 N/mm² at W/B 35% for
ordinary cement and 42.5 N/mm² at W/B 45% for blast furnace cement in the whole immersion experiment. In the partial contact experiment, the compressive strength was 30.9 N/mm² at W/B 65%. The chemical admixture used for concrete was a high-performance AE water reducing agent when the W/B was 35%, while an AE water reducing agent was used for other water binder ratios.

In order to avoid the effect of bleeding at the interface between concrete and reinforcing steel as much as possible, all specimens were vertically cast in the direction of 400 mm in length. The specimens were demolded 24 hours after casting and standard cured up to 28 days of age. After that, the surface coating material of an acrylic resin system was applied to four sides, and dried in indoor air for seven days, and after saturating with fresh water for another five days, soaking in brine water was begun. The reason why the surface coating material was applied to the four side surfaces was to suppress salt supply from the side surface of the specimen in the whole immersion experiment, and to suppress evaporation of water from the side surface of the specimen in the partial contact experiment.

### 3.3 Experimental method

#### 3.3.1 Chloride ion supply method

Figure 8 shows the immersion situation in salt water in the whole immersion experiment. In this experiment, the specimen was placed on a vinyl chloride pipe in an immersion water bath, with the surface coated with a surface coating material turned upside-down, and with the exposed surface turned from left to right. The brine was a 10% sodium chloride solution, and the drying and wetting were repeated for three days in immersion and four days in the drying cycle.

Figure 9 shows the contact situation with salt water in the partial contact experiment. In this experiment, the specimen was placed on a vinyl chloride pipe in a dry container, with the surface coated with a surface coating material turned from left to right, and with the exposed surface turned upside-down. The hollow rectangular plastic container (inner space size: 50 mm × 90 mm) was fixed at the center part of the 400 mm length of the specimen, and was filled with a 10% sodium chloride solution to be contacted with salt water only within the range of 50 mm. In both experiments, the specimen was placed in an indoor environment that was not affected by wind, rain, or solar radiation, but was affected by ambient temperature and humidity.
3.3.2 Measurement method of the half-cell potential and visual observation by dismantling

Figure 10 shows the method of measuring the half-cell potential in the whole immersion experiment. The experiment was measured at 15 points at intervals of 25 mm in the direction of 400 mm in the length just above the reinforcing bars on the specimen surface while using a lead reference electrode. The half-cell potential was measured at 0, 6, 8 and each cycle thereafter, when switching from immersion to drying. A visual observation of the existence of corrosion by disassembly of the specimen was carried out when the half-cell potential of any measuring point reached the lower value from -350 mV vs CSE, in which the occurrence probability of the corrosion was thought to be 90% or more in ASTM C876 (ASTM 2009). In this paper, the half-cell potential is expressed by subtracting 799 mV from the half-cell potential measured by a lead reference electrode and converting it to the half-cell potential for a saturated copper sulfate electrode (vs CSE).

Figures 11 and 12 show the measuring method of the half-cell potential in the partial contact experiment. In this experiment, the half-cell potential was automatically measured every 10 minutes from the start of contact with salt water on the specimen surface with the lead reference electrode embedded in the specimen. In the visual observation concerning the existence of corrosion by disassembly of the specimen, the disassembly time was decided appropriately after the change in the half-cell potential appeared.

In addition, in order to confirm the state of chloride ion penetration, the surface analysis by EPMA was carried out on the specimen after disassembly, and the chloride ion quantity distribution in the specimen cross section was obtained.
3.4 Experimental results
3.4.1 Results in the whole immersion experiment
Figures 13 and 14 show the measurement results of the half-cell potential in the whole immersion experiment. Figure 13 shows an example of the results of a specimen with W/B 35% using ordinary cement, which was disassembled in 11 cycles and visually observed. At 11 cycles, the most ignoble potential of the measurement point was -313 mV (vs CSE), and corrosion was observed 260 mm from the left edge of the specimen as shown in Fig. 15. In this specimen, the lowest half-cell potential was -346 mV (vs CSE) at six cycles, which was slightly more noble than -350 mV (vs CSE). Therefore, the changing-over time of the half-cell potential was waited for without dismantling, however since then it shifted higher, so was disassembled at the 11th cycle, and the occurrence of corrosion was observed.

As with this specimen, in the whole immersion experiment, corrosion sometimes occurred even in a condition higher than -350 mV (vs CSE) which is a half-cell potential as a standard for the corrosion of reinforcing steel in concrete according to ASTM criteria, and it was not possible to clarify when corrosion occurred.

On the other hand, Fig. 14 shows an example of the results of a specimen with W/B 45% using blast furnace cement. The specimen was disassembled in six cycles and visually observed. At the time of six cycles, the most ignoble potential was -447 mV (vs CSE), which was lower than -350 mV (vs CSE), and corrosion was observed at a position 60 mm from the edge of the specimen as shown in Fig. 16. In this specimen, rust liquid was propagated along the interface between coarse aggregate and mortar. This is because the maximum dimension of 13 mm of coarse aggregate was too large to cover 15 mm. Therefore, the water channel through which salt water easily passed was generated at the interface between coarse aggregate and mortar, and corrosion seemed to occur early in the experiment.

3.4.2 Results in the partial contact experiments
Figures 17 and 18 show the measurement results of the half-cell potential in the partial contact experiment. Fig. 17 shows the time course of the half-cell potentials of a single bar specimen with a cover of 20 mm, and a double rebar specimen with covers of 20 mm and 25 mm. In addition, Fig. 18 shows the time change course of the rebar potential around the time when a large change was observed in the half-cell potential of the rebar in the double rebar specimen in Fig. 17.

In the single rebar specimen, the half-cell potential of -170 mV (vs CSE) at 978 hours after the start of salt water contact suddenly decreased to -261 mV (vs CSE) at 983 hours after five hours. In the rebar with a cover of 20 mm, in the double rebar specimen, the half-cell potential

![Fig. 13 Measurement result of the half-cell potential in the whole immersion experiment (ordinary cement, W/B 35%).](image1)

![Fig. 14 Measurement result of the half-cell potential in the whole immersion experiment (blast furnace cement, W/B 45%).](image2)

![Fig. 15 Situation regarding corrosion occurrence on the steel bar surface (ordinary cement, W/B 35%).](image3)

![Fig. 16 Situation regarding corrosion occurrence on the steel bar surface (blast furnace cement, W/B 45%).](image4)
of -133 mV (vs CSE) at 382 hours dropped sharply to -264 mV (vs CSE) at 408 hours after 26 hours. On the other hand, in the rebar with a cover of 25 mm, there was little change as -134 mV (vs CSE) in 382 hours and -138 mV (vs CSE) in 408 hours.

As shown in Fig. 19, when the single rebar specimen was disassembled at 983 hours (five hours after the half-cell potential dropped), corrosion was observed on the rebar at a position of 202 mm from the left edge. In addition, as shown in Fig. 20, when the double rebar specimen was disassembled at 408 hours (26 hours after the half-cell potential dropped), in the rebar with a cover of 20 mm, corrosion was observed on the rebar at a position of 205 mm from the left edge. However, no corrosion was observed on the surface of the rebar with a cover of 25 mm. The results were similar for the other two specimens fabricated and tested under the same conditions (Horiguchi et al. 2006).

Figures 21 and 22 show the distribution of chloride ion concentration in concrete by EPMA analysis in the dismantled double rebar specimen. Figure 21 shows the EPMA image at the cut section in the middle part of the specimen in the longitudinal direction of the contact position with salt water. On the other hand, Fig. 22 shows the EPMA image at the cut plane horizontal to the longitudinal direction of the specimen, and 25 mm from the left of the image is the range of contact with salt water. From these images, it is found that chloride ions permeate almost uniformly in the depth direction in the width range of 50 mm (long edge direction) × length 90 mm (short side direction) contacting with salt water.

From the above, by constantly contacting salt water to a part of the surface of the specimen and locally permeating chloride ions into concrete, and by continuously measuring the half-cell potential of steel materials with a buried lead reference electrode, it was confirmed that the time when corrosion occurred in the steel material inside the concrete could be clearly ascertained. It was also considered that the chloride threshold value could be quantitatively evaluated by measuring the total chloride ion quantity at the steel surface position where corrosion occurred at this time.

4. Experiments to evaluate the chloride threshold value for corrosion initiation quantitatively

4.1 Outline of the experiment

In the experiment on the quantitative evaluation of the chloride threshold value for corrosion initiation, reinforced concrete specimens were manufactured with the type of cement and water binder ratio as factors. The time
of corrosion occurrence of steel materials was ascertained by the method of the above partial contact experiment, while the total chloride ion quantity at the reinforcing steel position was measured at the same time to quantify the chloride threshold value.

In this Section, some numerical values are shown for the total chloride concentration calculated from the measured total chloride ion amount, but to avoid confusion, the following are defined.

(a) Measured total chloride concentration: The total chloride concentration measured on the specimen, is expressed in the mass per cubic meter of concrete.

(b) Calculated total chloride concentration: The total chloride concentration obtained by calculating the surface salt content $C_0$ and the apparent diffusion coefficient $D$ in the Fick's diffusion equation [Eq. (1) below] by the least squares method from the measured total chloride concentration distribution.

(c) Total chloride concentration at the time of steel corrosion occurrence: Calculated total chloride concentration on the surface of steel at the time of steel corrosion occurrence.

(d) Chloride threshold value for corrosion initiation: The value obtained by statistically processing the total chloride concentration at the time of steel corrosion occurrence for specimens with the same binder and the same water binder ratio. The minimum value [left-hand value in Eq. (2)] is calculated by the average interval estimation shown in Eq. (2) with a confidence factor of 95% (Azuma et al. 1987).

$$C_{t(x)} = C_0 \left(1 - \text{erf} \left( \frac{x}{200D \cdot t} \right) \right) \quad \text{(1)}$$

where $C_{t(x)}$ is the total chloride concentration (kg/m³) at cover $x$ (m) and time $t$ (year), $C_0$ is the total chloride concentration on the concrete surface (kg/m³), erf is the error function, and $D$ is the apparent diffusion coefficient (m²/year).

$$\left[ \frac{x - \bar{x}}{\sqrt{n} \cdot \frac{u}{\sqrt{n}}} \right] - \frac{t_{n-1} \left( \alpha \right) \cdot u}{\sqrt{n}}$$

where $\bar{x}$ is the sample mean, $n$ is the sample number, $u$ is the sample variance, $1 - \alpha$ is the confidence coefficient (here 0.05), and $t_{n-1} \left( \alpha \right) u$ are the t-distributions with $n - 1$ degrees of freedom.

### 4.2 Specimen

The shape and dimensions of the specimen, and the arrangement of reinforcing bars and lead reference electrodes were the same as those shown in Figs. 6 and 7. The specimen size is $100 \times 100 \times 400$ mm, the maximum size of coarse aggregate is 13 mm, and the double bars of D19 of SD295A were placed with covers at 20 mm and 25 mm. A lead reference electrode with a diameter of 10 mm was also arranged in the same way.

Table 5 shows the mix proportions of an experimental specimen for quantitative evaluation of the chloride threshold value for corrosion initiation.

#### Table 5 Mix proportions of the specimen in an experiment on the quantitative evaluation of the chloride threshold value for corrosion initiation.

| Specimen No. | Gmax (mm) | Slump (cm) | Air volume (%) | W/B (%) | s/a (%) | Unit Amount (kg/m³) | Compressive Strength (N/mm²) | Dismantled specimen quantity |
|--------------|-----------|------------|----------------|---------|---------|---------------------|-----------------------------|-----------------------------|
| 65N          | 12.0      | 5.2        | 65             | 52      | 165     | 254                 | -                           | 974                         | 904                         | 2.55                       | 28.1                       | 7                           |
| 55N          | 11.0      | 5.0        | 55             | 50      | 160     | 291                 | -                           | 929                         | 933                         | 3.63                       | 49.7                       | 6                           |
| 45N          | 11.5      | 4.0        | 45             | 48      | 163     | 362                 | -                           | 858                         | 933                         | 3.63                       | 49.7                       | 6                           |
| 35N          | 14.0      | 4.9        | 35             | 46      | 156     | 446                 | -                           | 800                         | 941                         | 3.63                       | 65.4                       | 3                           |
| 65BB         | 14.5      | 4.6        | 65             | 52      | 171     | 132                 | 132                         | 938                         | 892                         | 3.63                       | 65.4                       | 3                           |
| 55BB         | 12.5      | 4.8        | 55             | 50      | 156     | 442                 | 142                         | 932                         | 943                         | 2.85                       | 34.5                       | 4                           |
| 45BB         | 12.5      | 4.3        | 45             | 50      | 159     | 177                 | 177                         | 898                         | 908                         | 3.55                       | 42.3                       | 3                           |
| 35BB         | 13.5      | 4.6        | 35             | 46      | 152     | 217                 | 217                         | 800                         | 943                         | 2.15                       | 60.9                       | 2                           |

Fig. 21 EPMA image of the double rebar specimen (center cross section of the specimen).

Fig. 22 EPMA image of the double rebar specimen (center longitudinal section of the specimen).
threshold value. The material used for the specimen was the same as that shown in Table 4. The concrete used in this study was composed of two types of cement: ordinary Portland cement and blast furnace cement. Blast furnace cement was a mixture of equal mass proportions of ordinary Portland cement and blast furnace slag fine powder. In Japan, this is equivalent to blast furnace cement type B, commonly called BB. There were two types of cement, four cases of W/B in 65, 55, 45, and 35%, and seven specimens for each case; 56 specimens in total were then prepared. The specimen symbol is a combination of the water binder ratio and the binder type, for example, a normal concrete cement specimen with W/B 65% is expressed as 65N, and a blast furnace cement concrete specimen with W/B 55% is expressed as 55BB. To identify the individual specimens, a serial number was added after the specimen symbol, such as 65N-1, 55BB-2.

The method of casting the specimen, curing method, surface covering of four side surfaces, etc. were all the same as those in the above partial contact experiment. The compressive strength of the ordinary cement specimen after 28 days of standard curing was 28.1, 39.9, 49.7 and 65.4 N/mm², when W/B was 65, 55, 45 and 35%, respectively. In the same way, the compressive strength of the blast furnace cement specimen was 29.4, 34.5, 42.3 and 60.9 N/mm², respectively.

Specimens were manufactured as seven bodies in each case; some specimens were still continuing the experiment five years from the start of the exposure. In addition, for some specimens, the corrosion occurrence time was not clearly captured due to malfunction of the lead reference electrode. Therefore, for ordinary cement specimens, the number of specimens from which significant data could be obtained was seven bodies when W/B was 65% and 55%, six bodies when W/B was 45%, and three bodies when W/B was 35%. In the case of blast furnace slag cement specimens, the number of specimens from which significant data could be obtained was five bodies when W/B was 65%, four bodies when W/B was 55%, three bodies when W/B was 45%, and two bodies when W/B was 35%.

4.3 Experimental method
4.3.1 Chloride ion supply method
The salinity supply method and the exposure environment of the specimen were the same as those in the partial contact experiment shown in Fig. 9.

4.3.2 Measurement method of the half-cell potential and visual observation by dismantling
The method of measuring the half-cell potential was the same as that of the partial contact experiment shown in Figs. 11 and 12. A visual observation of corrosion due to disassembly of the specimen was carried out after the change of the half-cell potential of the rebar when a cover of 20 mm appeared.

4.3.3 Salinity measuring method
Figure 23 shows the measured position of total chloride concentration. The measurement of total chloride ion quantity was carried out by cutting out a slice of concrete in the depth direction. The size of the slice was 25 mm wide (in the long side direction), 70 mm long (in the short edge direction), 5 mm thick (in the depth direction), and was collected at four or five places in the depth direction. At this time, the sampling depth of the slices was set at four points at average depths of 2.5, 12.5, 22.5 and 27.5 mm from the specimen surface. In the case of five points, an additional point with an average depth of 17.5 mm was also measured. The range of 25 mm in the width (in the long side direction) of the slice was selected for each test piece so that the corrosion position of the rebar with a cover of 20 mm was included in the width. The measurement method of the total chloride ion quantity was according to JIS A 1154 "Method for testing chloride ion contained in cured concrete". The total amount of chloride ions was measured by cutting out and grinding a slice of concrete within 48 hours after disassembling the specimen.

4.4 Experimental results
4.4.1 Half-cell potential and corrosion occurrence situation
Figures 24 and 25 show the time course of the half-cell potential. Regardless of the type of binder and the water binder ratio, the half-cell potential of the rebar with a cover of 20 mm in all specimens dropped sharply at the time of corrosion occurrence. On the other hand, no sudden decrease in the half-cell potential was observed in the rebar buried in the same specimen with a cover of 25 mm. Here, the time when the stable potential suddenly dropped by 10 mV was evaluated as the time when corrosion started.

Tables 6 and 7 show the time from the start of the experiment to the lowering of the half-cell potential of the rebar with a cover of 20 mm, the time from the lowering of the half-cell potential to the dismantling, and the corrosion area. The corrosion was generated in all rebars with a cover of 20 mm in which the half-cell potential lowered, and the corrosion area was 2 to 176 mm². On the other hand, no corrosion was observed in any of the rebars with a cover of 25 mm without a decrease in
half-cell potential. Among these specimens, 35N-2, 45BB-1, and 35BB-2 were disassembled and observed visually after a relatively long time of 3319, 2644, and 3123 hours, respectively, from the lowering of the half-cell potential to disassembly. However, the half-cell potential of the rebar with a cover of 25 mm did not decrease, and corrosion was not observed. In addition, the change of the half-cell potential when the potential dropped and disassembled was -132 to -238 mV (vs CSE) in 35N-2, -141 to -253 mV (vs CSE) in 45BB-1, and -130 to -226 mV (vs CSE) in 35BB-2. The half-cell potential changed more in the nobler potential than -350 mV (vs CSE), but corrosion occurred in these specimens. The corrosion areas of these specimens were 155, 176 and 67 mm² respectively, so they were not extremely large compared with the other specimen corrosion areas of 2 to 136 mm². This is probably due to the fact that the corrosion rate was slow because the half-cell potential did not decrease below -350 mV (vs CSE).

4.4.2 Measured total chloride concentration distribution

Figures 26 and 27 show the distribution of measured total chloride concentration in the depth direction. Figure 26 shows the measured total chloride concentration distribution of ordinary cement specimens with W/B 65%, and Fig. 27 shows that of blast furnace cement specimens with W/B 65%. There was a tendency for dispersion in the concentration distribution of blast furnace cement specimens compared with that of ordinary cement specimens.

4.4.3 Surface total chloride concentration and apparent diffusion coefficient

Tables 6 and 7 also show the surface total chloride concentration $C_s$ and the apparent diffusion coefficient $D$ for each specimen. These values were calculated by the least squares method from the measured total chloride concentration distribution shown in Figs. 26 and 27. In the ordinary cement specimen, the lower the water binder ratio (65, 55, 45 and 35%), the more the average of the surface total chloride concentration $C_s$ increased (20.3, 21.9, 30.1 and 43.5 kg/m³), and the average of the apparent diffusion coefficient $D$ became smaller (4.65, 3.13, 1.36 and 0.25 cm²/year). On the other hand, this tendency was not seen in the blast furnace cement specimens, which did not have a definite relationship between the water binder ratio (65, 55, 45 and 35%) and the average of total surface chloride ion concentration $C_s$ (28.8, 38.7, 27.9 and 17.8 kg/m³). While the apparent diffusion coefficient $D$ (0.54, 0.22, 0.26 and 0.29 cm²/year) was a little larger at 65% of the water binder.
It was the same value degree in other water binder ratios.

4.4.4 Total chloride concentration at the time of steel corrosion occurrence

Tables 6 and 7 also show the total chloride concentration at the time of steel corrosion occurrence for each specimen. The total chloride concentration shown here is the calculated total chloride concentration in Eq. (1), obtained by using the total surface chloride ion concentration $C_0$ and the apparent diffusion coefficient $D$ for each specimen, and where the cover $x$ is 20 mm and the time $t$ is the time when the half-cell potential has dropped. The time dependence of the total surface chloride ion concentration $C_0$ and the apparent diffusion coefficient $D$ was not considered, because the time from the half-cell potential drop to the dismantling of the specimen was much shorter than that from the start to the dismantling.

4.4.4 Chloride threshold value for corrosion initiation

Tables 8 and Figs. 28 to 31 show the range and average of the total chloride concentration at the time of steel corrosion occurrence, and chloride threshold value for corrosion initiation. The total chloride concentration at the time of steel corrosion occurrence was the value calculated from the total chloride concentration distribution measured at the time of specimen disassembly, and these concentration values were different from each specimen. Therefore, the chloride threshold value of the specimen with the same binder and the same water binder ratio was the value statistically calculated from the total chloride concentration at the time of steel corrosion occurrence in each specimen by Eq. (2). In this case, it was the minimum value calculated by the equation of the average interval estimation with a confidence factor of 95%.

Corrosion areas of 65N-2, 55N-6 have no measurement data.
two, and the value becomes negative when the statistical treatment is carried out, a significant result is not obtained. Therefore, the chloride threshold value is not shown.

Figure 28 shows the relationship between the unit amount of the binder and the chloride threshold value for corrosion initiation in an ordinary cement specimen. As the unit amount of the binder increases to 254, 291, 362, and 446 kg/m³, the chloride threshold value increases to 1.6, 2.5, 3.0 and 3.6 kg/m³, respectively. In addition, the simple average of the total chloride concentration at the time of steel corrosion occurrence is 2.3, 3.3, 3.7 and 5.1 kg/m³, respectively. This is considered to be due to an increase in the amount of solid phase chlorides such as Friedel's salt. It is considered that the amount of hydroxide ions dissolved in the pore solution increases as the amount of binder increases. Therefore, assuming that the molar ratio of chloride ion concentration to hydroxide ion concentration when corrosion occurs is constant, it is considered that the chloride ion concentration increases as the amount of binder increases. This indicates that the unit amount of the binder is one of the important factors in determining the chloride threshold value. Incidentally, if this is expressed as the notation for mass % of the binder, it becomes 0.63, 0.85, 0.84, and 0.80 mass % of the binder for 254, 291, 362, and 446 kg/m³ of the unit amount of the binder, respectively, and becomes about 0.8 mass % of the binder, although it is slightly small for 254 kg/m³ of the unit amount of the binder. This value is in the range of about 0.3 to 1.8 mass % of the binder shown in the previous study, and is almost the middle value, so it does not seem to be a peculiar result even in comparison with the result of the previous study.

Figure 29 shows the relationship between the water binder ratio and the chloride threshold value for corrosion initiation in the ordinary cement specimen. At the water binder ratios of 65, 55, 45, and 35%, the chloride threshold value was 1.6, 2.5, 3.0, and 3.6 kg/m³, respectively, and the threshold value increased as the water binder ratio decreased. This is because the unit amount of the binder is 254, 291, 362, and 446 kg/m³ when the water binder ratios are 65, 55, 45, and 35%, respectively. The chloride threshold value is mainly determined by the unit amount of the binder as described above. In an ordinary concrete, there is not much difference in the unit amount of water due to the change in the water binder ratio, but the unit amount of the binder increases or decreases depending on the water binder ratio. So, such a relationship can also be found in the water binder ratio and the chloride threshold value.

Figure 30 shows the relationship between the unit amount of the binder and the chloride threshold value for corrosion initiation in the blast furnace cement specimen. The unit amount of the binder is 264, 284, and 354 kg/m³ and the chloride threshold value is 1.6, 1.2, and 1.9 kg/m³. In addition, the simple average of the total chloride concentration shown in the previous study is considered to be 1.8 mass % of the binder, so it does not seem to be a peculiar result even in comparison with the result of the previous study.

| Specimen No. | Time to potential drop (h) | Time from potential drop to disassembly (h) | Corrosion area (mm²) | Total chloride concentration on the concrete surface $C_0$ (kg/m³) | Apparent diffusion coefficient $D$ (cm²/year) | Total chloride concentration at the time of steel corrosion occurrence (kg/m³) |
|--------------|----------------------------|--------------------------------------------|----------------------|---------------------------------------------------------------|---------------------------------------------|---------------------------------------------------------------|
| 65BB-1       | 19062                      | 97                                         | 27.0                 | Ave. 28.8                                                      | 0.34                                        | Ave. 2.78                                                      |
| 65BB-2       | 9253                       | 57                                         | 86.4                 | 1.05                                                          | 0.30                                        | 2.45                                                          |
| 65BB-3       | 16929                      | 41                                         | 18.7                 | 0.60                                                          | 0.42                                        | 2.25                                                          |
| 65BB-4       | 8657                       | 124                                        | 39.9                 | 0.42                                                          | 2.47                                        |                                                               |
| 65BB-5       | 18799                      | 22                                         | 3.28                 |                                                               |                                              |                                                               |
| 55BB-1       | 20783                      | 49                                         | 3.97                 | Ave. 38.7                                                      | 0.23                                        | Ave. 2.87                                                      |
| 55BB-2       | 37113                      | 544                                        | 74.3                 | 0.16                                                          | Ave. 0.22                                   | 4.01                                                          |
| 55BB-3       | 37177                      | 480                                        | 37.2                 | 0.20                                                          | Ave. 0.26                                   |                                                               |
| 55BB-4       | 15212                      | 51                                         | 45.9                 | 0.27                                                          |                                             |                                                               |
| 45BB-1       | 37462                      | 2644                                       | 176.0                | Ave. 27.9                                                      | 0.52                                        | Ave. 4.12                                                      |
| 45BB-2       | 46118                      | 82                                         | 30.9                 | 0.12                                                          | Ave. 0.26                                   | 3.08                                                          |
| 45BB-3       | 45596                      | 101                                        | 64.0                 | 0.14                                                          |                                             | 3.01                                                          |
| 35BB-1       | 37381                      | 370                                        | 136.5                | Ave. 21.8                                                      | 0.21                                        | Ave. 3.38                                                      |
| 35BB-2       | 29565                      | 3123                                       | 67.3                 | 0.36                                                          | Ave. 0.29                                   | 2.09                                                          |

Corrosion area of 65BB-1 has no measurement data.

Table 7 Experimental results in the blast furnace cement specimen.
concentration at the time of steel corrosion occurrence was 2.8, 2.8 and 3.4 kg/m³, respectively. There is no clear indication that the chloride threshold value increases with the unit amount of the binder, as in ordinary cement specimens. However, in the notation for mass % of the binder, it becomes 0.52, 0.43, and 0.59 mass % of the binder, respectively, and becomes approximately 0.5 mass % of the binder. On the other hand, it is about 0.8 mass % of the binder in the case of ordinary cement. It is considered that the increase of the chloride threshold value does not clearly appear with the increase of the unit amount of binder compared with ordinary cement. This is because, assuming that the molar ratio of chloride ion concentration to the hydroxide ion concentration in the pore solution at the time of corrosion occurrence is constant, the pH of the pore solution in the blast furnace cement is lower than that in ordinary cement, so that the amount of chloride ions dissolved in the pore solution is also smaller in the blast furnace cement.

Figure 31 shows the relationship between the water binder ratio and the chloride threshold value for corrosion initiation in the blast furnace cement specimen. At the water binder ratios of 65, 55, and 45%, the chloride threshold value for corrosion initiation was 1.6, 1.2, and 1.9 kg/m³, respectively. As described above, in the blast furnace cement specimens, there was no clear relationship between the unit amount of the binder and the chloride threshold values for corrosion initiation. Consequently, there was no clear relationship between the water binder ratio and the chloride threshold values either. However, it was confirmed that the chloride threshold values for corrosion initiation in blast furnace cement specimens was more than 1.2 kg/m³ as shown in the Standard Specifications for Concrete Structures (JSCE 2007).

5. Conclusions

The purpose of this study is to develop a method to evaluate accurately the chloride threshold value, and to determine the threshold value quantitatively by this experimental method. The chloride threshold value in this paper is not a simple average of the total chloride concentration at the time of steel corrosion occurrence obtained with multiple specimens of the same mix proportion, but is the minimum value when statistical processing of the average interval estimation is performed with a confidence coefficient of 95%. The following are the

| W/B and Cement types | Unit amount of binder (kg/m³) | Range and average of total chloride concentration at the time of steel corrosion occurrence (kg/m³) | Chloride threshold value for corrosion initiation (kg/m³) |
|----------------------|-------------------------------|--------------------------------------------------------------------------------------------------|----------------------------------------------------------|
|                      | Min | Max  | Average |                                           |                                                         |
| 65N                  | 254 | 1.25 | 3.57    | 2.29                                       | 1.59                                                    |
| 55N                  | 291 | 2.37 | 4.53    | 3.27                                       | 2.47                                                    |
| 45N                  | 362 | 2.66 | 4.72    | 3.74                                       | 2.98                                                    |
| 35N                  | 446 | 4.58 | 5.81    | 5.13                                       | 3.57                                                    |
| 65BB                 | 264 | 2.04 | 4.47    | 2.78                                       | 1.57                                                    |
| 55BB                 | 284 | 1.79 | 4.01    | 2.80                                       | 1.21                                                    |
| 45BB                 | 354 | 3.01 | 4.12    | 3.40                                       | 1.85                                                    |
| 35BB                 | 434 | 2.09 | 3.38    | 2.74                                       | —                                                       |

The chloride threshold value for corrosion initiation of 35 BB was a negative value in calculation.
findings obtained through this study.

1) The corrosion occurrence time of reinforcing steel in reinforced concrete could be evaluated by continuously measuring the half-cell potential with a lead reference electrode embedded in the specimens while locally supplying chloride ions from the surface of the specimens to the inside.

2) The chloride threshold value of concrete using ordinary cement was 1.6, 2.5, 3.0, and 3.6 kg/m³, respectively, when the unit amount of the binder was 254, 291, 362, and 446 kg/m³, and it increased with the unit amount of the binder. In the notation for mass % of the binder, this became 0.63, 0.85, 0.84, and 0.80 mass % of the binder, respectively.

3) The chloride threshold value of concrete using blast furnace cement was 1.6, 1.2, and 1.9 kg/m³, respectively, when the unit amount of the binder was 264, 284, and 354 kg/m³. There was no clear indication that the chloride threshold value increased with the unit amount of the binder, as in ordinary cement concrete. But in the notation for mass % of the binder, it became 0.52, 0.43, and 0.59 mass % of the binder, respectively. Since the pH in the pore solution of concrete using blast furnace cement is lower than that of concrete using ordinary cement and assuming that the molar ratio of the chloride ion concentration to the hydroxide ion concentration in the pore solution at the time of corrosion occurrence is constant, the amount of chloride ions dissolved in the pore solution is also smaller in the blast furnace cement. Therefore, it is thought that the chloride threshold value of the blast furnace cement concrete also becomes lower than that of ordinary cement concrete.

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