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Feasibility Study of Nondestructive Diagnostic Method for Chlorine in Concrete by Compact Neutron Source and PGA

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Abstract

As a nondestructive measurement of chloride ion distribution in concrete is important from the viewpoint of preventive maintenance against chloride attack causing deterioration of many concrete structures, a diagnostic technique of a non-destructive measurement method using a neutron-captured prompt gamma-ray analysis (PGA) is being developed. As the first step of development, the γ -ray sensitivities of mortar samples with different chloride ion concentrations were determined experimentally by PGA using the RIKEN accelerator-driven compact neutron source. The results showed that the present detection system was sensitive to a chloride ion concentration of 1 kg/m³, which is lower than the marginal chloride ion concentration of 1.2 - 2.5 kg/m³ to incur corrosion. The time of flight measurement technique with pulsed neutrons was applied concerning the depth profile of chloride ion distribution in concrete.

1. Introduction

It is important to develop a reliable nondestructive diagnostic technique that can be used on site, in order to maintain concrete structures such as roads and bridges. In Japan, concrete structures, in particular, bridge, are exposed to sea breeze from coastal areas and also to chloride contained in de-icing agents spread in cold and mountainous areas. The chloride attack due to infiltration of chloride ions (Cl⁻) into concrete is a serious issue. Corrosion starts once the chloride ion concentration around the rebar in the concrete exceeds the marginal chloride ion concentration of 1.2 - 2.5 kg/m³, which depends on the water cement ratio (JSCE 2012). Then it is important to diagnose chloride ion distribution for the maintenance of concrete structures. In particular, depth profile of concrete structure from the surface to a region nearby rebar is critical.

Although there are existing measurement methods of chloride ion distribution such as fluorescent X-ray

analysis (Kanada *et al.* 2006), electron beam micro-analyzers (JSCE 2005), and potentiometric titration methods in JIS A 1154 (JIS 2012), they need the core samples to be collected from the structures to be tested. In addition, it takes time and cost for premeasurement processing. Thus the above methods are not appropriate to cover the whole area required to be tested. Moreover, once core samples are taken from the subjected locations, the sampling locations are no longer valid for aging monitoring, and may cause some damage to the structures.

In order to overcome such issues, a nondestructive method is desirable. Although an existing non-destructive method on the concrete surface by near-infrared spectroscopy (Kanada *et al.* 2005) is useful to carry out measurements inside the concrete by inserting the fiber sensor through a small hole, it requires holes to be drilled. Therefore, research and development on the measurement of chloride ion distribution inside concrete using Neutron-capture Prompt Gamma-ray Analysis (NPGA) was initiated, aiming at practical use of nondestructive diagnostic method without core sampling and drilling holes. Here, the authors have tried to demonstrate the feasibility of NPGA with a compact pulsed neutron source for detection of chlorine concentration.

The experiments to measure the chloride ion concentration was conducted using the RIKEN Accelerator-driven compact Neutron Source, RANS (Yamagata *et al.* 2015; Otake *et al.* 2017; Wakabayashi *et al.* 2018; Ikeda *et al.* 2016), which is a neutron source that has been actively operated to develop nondestructive testing technologies (Ikeda *et al.* 2017; Taketani *et al.* 2017; Yoshimura *et al.* 2019), and a portable pulse neutron source for onsite use. To obtain the NPGA sensitivity to the chloride ion concentration by using pulsed thermal neutrons from RANS, mortar specimens with chloride ion concentrations ranging from 0.36 to 5.1 kg/m³ were prepared, and a method to estimate the chloride ion distribution

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using the neutron time of flight with pulsed fast and thermal neutrons of RANS was also studied. This paper describes the applicability of the system as a nondestructive technique.

Yamada *et al.* (2009) have studied the distribution of chlorine detection using NPGA. The differences between their study and the current study at RANS are as follows: (1) The above study by Yamada *et al.* (2009) was performed at nuclear reactor JRR-3 with high neutron flux and current neutron, whereas the current study was performed with an accelerator-driven compact neutron source with low neutron flux and pulsed neutrons, including fast neutrons. (2) The above study was focused relatively nearer to surface, 25 mm depth, for example, while the current study is aimed at deeper areas using not only thermal neutrons but also fast neutrons. (3) In the above study, the depth profile was deduced utilizing the γ -rays of calcium-40 and chlorine-35, while the current study employs two different γ -rays energies from chlorine-35.

Note that the term of “chloride ion” is used in the descriptions associated with chloride ion distribution or concentration, “chloride” is used to indicate the chemical form, and “chlorine” is used when referring to elemental chlorine for the detection by NPGA.

2. Methods for chlorine detection

The method for chlorine detection makes use of a neutron source, prompt γ -ray detection, and the samples with chlorine.

2.1 RIKEN Accelerator-driven compact Neutron Source, RANS

Figure 1 shows the structure of RANS as side and top views. RANS accelerates protons to 7 MeV with a linear accelerator, and protons are injected to a beryllium target for neutron production. Neutrons are slowed down to lower energy by a 40 mm thick polyethylene (PE) moderator and the neutrons are extracted from the target station to the specimen placed in each experimental setup. The cross section of the neutron beam was adjusted to less than $160 \times 160 \text{ mm}^2$ by using PE colli-

Fig. 1 Schematic structure of RANS.

imators, a slightly tapered collimator made of B_4C powder, a boronated polyethylene (BPE) block, and a compact quadrilateral slit made of sintered B_4C . As shown in Fig. 2, the thermal neutron flux is approximately 10^4 neutrons/cm²/s at a position 5 m from the beryllium target when the proton beam current is 100 μA . In this study, the experiment was conducted when the average beam current was in range from 36 to 50 μA . The neutron beam had $50 \times 50 \text{ mm}^2$ cross section and the specimen was positioned 3.5 m from the beryllium target.

2.2 Neutron-captured prompt γ -ray analysis (NPGA) technique

NPGA is a method to identify elements by detecting γ -rays emitted from excited states that are produced via neutron capture reactions of the target nucleus. A conceptual diagram of the prompt γ -ray emission is shown in Fig. 3. Since this prompt γ -ray energy and its associated intensity are inherent to nuclei, multiple elements in a sample can be identified simultaneously. For example, there are associated major γ -rays of 517, 786, 788, 1165, 1951, and 6111 keV with intensity ratios of 22.8, 10.3, 16.3, 26.8, 19.1, 19.8%, respectively, in the neutron capture reaction of the nucleus of interest in this study, chlorine-35 (^{35}Cl). Regarding high penetrability of the neutrons, it was thought that information of capture reactions in depth of thick concrete could be obtained. Gamma-rays with 1 MeV or higher energies could penetrate concrete of more than ten centimeter thickness. Both neutron and γ -ray characteristics enable detection of chlorine in concrete, because the cross section of capture reaction, the $^{35}\text{Cl}(n, \gamma)^{36}\text{Cl}$, is large as shown in Table 1, and because of the high energies such as 1165, 1951, and 6111 keV of the associated major γ -rays from ^{36}Cl . Table 1 shows the mix proportion of main elements contained in ordinary concrete in weight percent with reference to the elemental ratios of ordinary concrete in Monte Carlo simulation code PHITS (Sato *et al.* 2018) and the capture reaction cross sections for the thermal neutron of 25 meV taken from

Fig. 2 RANS neutron spectrum at 5 m from the beryllium target with 40 mm thick polyethylene moderator.

Table 1 Weight ratio of main elements contained in ordinary concrete and the cross sections of thermal neutron capture.

Element	H	C	O	Mg	Al	Si	K	Ca	Fe	Cl
Cross section for thermal neutron (barn)	0.332	0.00386	0.00019	0.0630	0.230	0.165	2.1	0.429	2.565	33.15
Mix proportion (wt.%)	1.03	0.1	54.46	0.22	3.48	34.6	0.22	4.46	1.43	—

Table 2 Details of the specimens used in the experiment.

Type	Size	Chloride ion concentration	Content of ^{35}Cl
Polyvinylidene chloride (PVDC) film	$30 \times 30 \text{ mm}^2$ ($t = 0.10 \text{ mm}$) $20 \times 20 \text{ mm}^2$ ($t = 0.22 \text{ mm}$)	—	Approximately 85 mg
Cement paste	$76 \times 57 \text{ mm}^2$ ($t = 50 \text{ mm}$)	0, 6.1, 12 kg/m^3	0, 580, 1100 mg within $50 \times 50 \text{ mm}^2$
Mortar	$40 \times 40 \text{ mm}^2$ ($t = 40 \text{ mm}$)	0, 0.36, 0.56, 1.1, 3.1, 5.1 kg/m^3	0, 17, 27, 55, 150, 250 mg
	$40 \times 40 \text{ mm}^2$ ($t = 10 \text{ mm}$)		0, 4, 7, 14, 37, 62 mg

JENDL database (Shibata *et al.* 2011). To show the degree of reaction probability, the unit of cross section given is the barn, which corresponds to 10^{-24} cm^2 . The value shown in **Table 1** includes a stable isotope. Chlorine (Cl) has stable isotopes of ^{35}Cl (natural abundance ratio 75.77%, capture cross section 43.6 barn) and ^{37}Cl (24.23%, 0.0433 barn). The capture cross section for an element mostly relies on ^{35}Cl .

2.3 Specimens and experimental setups

The specimens for experiment were i) films of polyvinylidene chloride (PVDC; $\text{C}_2\text{H}_2\text{Cl}_2$), which are commercially available plastic wraps, ii) cement paste bricks containing sodium chloride (NaCl), and iii) bricks made of mortar. **Table 2** shows the sizes, concentrations of chloride ion, and weights of ^{35}Cl deduced from the sizes and concentrations of the specimens. The cement paste brick was a mixture of ordinary Portland cement and water in a weight ratio of 1:1. The type of cement was also ordinary Portland cement for the mortar brick. In this study, the PVDC film of about 100 μm in thickness, containing a small amount of chlorine, was used to identify the limit of detection of chlorine by NPGA.

Figure 4 shows a schematic diagram of the experimental setup. The neutron beam with a cross section of $50 \times 50 \text{ mm}^2$ was extracted through the neutron guides N0 and N1 to the sample position placed at 3.5 m from the beryllium target. The γ -ray spectrum measurements were carried out by using two Ge detectors, placed

perpendicularly to the direction of the neutron beam extracted from the RANS target station. The distance from the beam axis center to the surface of the Ge detectors was about 75 mm. Lead bricks and LiF tiles were also placed around the Ge detectors for shielding against γ -rays and neutrons, respectively.

3. Sensitivity of experimental setup for chlorine detection

3.1 Chlorine detection in mortar specimens

Figure 5 shows the measured γ -ray energy spectrum of the PVDC film. The measurement time was 10 minutes. The γ -ray energy (keV/ch) and the counts per channel (Counts/ch) are plotted on the horizontal axis on the vertical axis, respectively. With this setting, observation of the γ -rays (517, 786+788, 1165 keV) derived from the neutron capture reaction (hereinafter referred to as ^{35}Cl -derived) for detection of chlorine was confirmed although statistics were insufficient. The amount of ^{35}Cl in this film is approximately 85 mg, which is almost

Fig. 3 Conceptual diagram of prompt γ -ray.

Fig. 4 Schematic diagram of experimental setup.

equivalent to the mortar specimen with chloride ion concentration of 1.7 kg/m^3 .

Next, measurements were undertaken for the cement paste bricks with chloride ion concentrations of 0, 6.1 and 12 kg/m^3 . **Figures 6(a) to 6(c)** illustrate the γ -ray spectra obtained by 10 minutes measurements for cement paste bricks with chloride ion concentration of 12 kg/m^3 , 6.1 kg/m^3 and 0 kg/m^3 . ^{35}Cl -derived γ -rays of 517, 786+788 and 1165 keV are observed in the spectra for 12 kg/m^3 and 6.1 kg/m^3 bricks. These peaks were not observed for the bricks without chlorine.

In addition to the measurements with the cement paste bricks, mortar specimens with contents close to ordinary concrete in use, were performed for chloride ion concentrations of 0.36, 0.51, 1.1, 3.1 and 5.1 kg/m^3 that were given by the potentiometric titration method of JIS A 1154 (JIS 2012). **Figure 7** shows the γ -ray energy spectra around 1951 keV as the representative ^{35}Cl -derived γ -ray measured corresponding to the specimens. Here, the count rate R_γ of each ^{35}Cl -derived γ -ray is defined as:

$$R_\gamma \text{ (counts/s)} = A/t/\varepsilon_\gamma/I_\gamma \quad (1)$$

Fig. 5 Gamma-ray spectrum obtained by irradiating a PVDC film.

Fig. 6 Gamma-ray spectra obtained by irradiating cement paste bricks.

Table 3 Intensity ratios of prompt γ -rays in neutron capture reaction and detection efficiency in each measurement.

Gamma-ray Energy (keV)	517	786	788	1165	1951	6111
Intensity ratio, I_γ (%)	22.8	10.3	16.3	26.8	19.1	19.8
Detection efficiency, ε_γ (%)	0.31	0.21	0.21	0.14	0.084	0.027

where A is the γ -ray peak counts, t is the measurement time (s), ε_γ is the γ -ray detection efficiency where the distance between the Ge detector and γ -ray point source is 75 mm and I_γ is the γ -ray intensity ratio when neutron capture of ^{35}Cl occurs as mentioned in Section 2.1. The values of ε_γ and I_γ are summarized in **Table 3**. R_γ for each case was normalized by the proton beam intensity.

Figure 8 shows the correlation between the chloride ion concentration and R_γ of each corresponding ^{35}Cl -derived γ -ray. Only statistical errors of R_γ are given

Fig. 7 Gamma-ray spectrum obtained by irradiating mortar specimens.

on the data. This result shows good linearity down to 0.36 kg/m^3 between R_γ for 517, 786+788, 1165, and 1951 keV and the chloride ion concentrations given in Section 2.3, except for 6111 keV, which was not measured for specimens with chloride ion concentrations of less than 1.1 kg/m^3 due to low detection efficiency. From the detection of ^{35}Cl -derived γ -rays down to 0.36 kg/m^3 and their linearity, it is clearly demonstrated that the system of NPGA under the current conditions can detect ^{35}Cl -derived γ -rays in the chloride ion concentration down to 0.36 kg/m^3 , and that it is possible to determine the chloride ion concentration with about 5% error using the linearity.

3.2 Depth index by γ -ray intensity ratio

This experiment was aimed at identifying the sensitivity of ^{35}Cl detection depending on depth of concrete by using the RANS neutrons (spectrum shown in Fig. 2). A PVDC film of size $20 \times 20 \text{ mm}^2$ and 0.22 mm in thickness was placed on rear surface of the mortar bricks with 10 mm, 20 mm, and 50 mm in thickness without NaCl. Mortar specimens of $40 \times 40 \text{ mm}^2$ and 40 mm in thickness with chloride ion concentration of 5.1 kg/m^3 were placed behind mortar bricks of 50 mm and 130 mm thicknesses without NaCl. Figure 9 illustrates the experimental configurations of the specimens. R_γ values as defined in Section 3.1 were derived from the measured γ -ray spectra of the different configurations shown in Fig. 9. Figure 10 shows the correlation of R_γ and the thickness of the mortar bricks. The vertical bars associated with R_γ are statistical errors. The result shows that the sensitivity of chlorine detection decreases with the mortar thickness. This is understandable due to the neutron flux attenuation. Because the position of the Ge detector respect to PCDV films or mortar specimens was always same, the ^{35}Cl -derived γ -rays from specimens were not attenuated.

Fig. 8 Correlation between count rate and chloride ion concentration (measured values).

3.3 Detection of chlorine in sample for an actual bridge

In order to confirm the feasibility of the system studied, a part of the sample from a girder section of an actual bridge that was damaged by chloride ion, was measured. A view of the whole bridge is shown in Fig. 11(A), a section of the girder in Fig. 11(B), a sampling position in the girder in Fig. 11(C) and the γ -ray detection system of the sample in Fig. 11(D). Rough dimensions are also indicated. Figure 12 shows the γ -ray spectrum measured for five minutes, which was in the same time range of the sample measurements. It clearly shows the ^{35}Cl -derived γ -rays. The R_γ for the 1165 keV in the spectrum of the bridge sample was compared with the R_γ values of the cement paste bricks with chloride ion concentrations of 6.1 kg/m^3 and 12 kg/m^3 . The derived R_γ of 2731 for the bridge is in between the R_γ of 1497 for the cement paste brick with chloride ion concentration of 6.1 kg/m^3 and the R_γ of 3558 for the cement paste brick with chloride ion concentration of 12 kg/m^3 . It is estimated that the chloride ion concentration of this bridge should be in a range between 6.1 and 12 kg/m^3 .

Fig. 9 Measurement of the depth reached by the neutrons.

Fig. 10 Correlation diagram between the thickness of mortar specimen and R_γ .

However, the volume of the bridge sample, which was approximately $100 \times 70 \times 100 \text{ mm}^3$, was quite large compared to that of the cement paste brick, which was $76 \times 57 \times 50 \text{ mm}^3$. As the penetration probabilities of neutrons and γ -rays depend on the types of aggregates, the R_γ values between 6.1 to 12 kg/m^3 is only an index in this case and it is not sufficient to quantify the chloride ion concentration yet. In the future, the authors aim to quantify the chloride ion concentration with help of simulation of neutron transport and γ -ray attenuation in concrete.

4. Results and discussion

4.1 Detection sensitivity for chloride ion concentration

From the correlation between the chloride ion concentration (horizontal axis) and R_γ (vertical axis) shown in Fig. 8, the experiments indicate that it is feasible with the current experimental setup to detect chloride ion concentration of at least 0.36 kg/m^3 with about 5% error using the linearity shown in Section 3.1, with about 20% statistical error. Note that the experimental condi-

Fig. 11 Photograph of a real bridge girder under chloride attack near the coast and the samples taken.

Fig. 12 Gamma-ray spectrum obtained by irradiating actual bridge sample.

tions were, i) a thermal neutron flux of about 4×10^3 neutrons/cm²/s with a proton beam current of about 40 μA , ii) a mortar specimen with a thickness of 40 mm, and iii) a distance of 75 mm from the center of specimen to the window of Ge detector. The concentration of 0.36 kg/m^3 is presumed to be sufficiently low compared to the marginal chloride ion concentration of 1.2 - 2.5 kg/m^3 . Therefore, there is a possibility that the degradation condition could be diagnosed before the corrosion occurs. However, when chlorine locating at depths greater than 50 mm from the surface is measured, the amount of neutron flux decreases to about one third as shown in Fig. 10, showing that the detection sensitivity becomes low by a factor of three. From simple calculation, the sensitivity limit is estimated to be about 1.1 kg/m^3 as long as only neutron attenuation is considered.

4.2 Estimation method of chloride ion distribution toward the depth direction

(1) Attenuation of γ -rays by concrete

For the mortar specimens with chloride ion concentrations of 5.1 kg/m^3 and 3.1 kg/m^3 , the ratios of R_γ for 517, 788, and 1165 keV with respect to that for 1951 keV are shown in Fig. 13. At the same chloride ion concentration, R_γ decreases with decreasing γ -ray energy. This can be considered to be because the mortar specimen has a thickness of 40 mm, which leads the variance of attenuation ratio in the specimen due to the energy of γ -rays. The bars with the experimental values represent statistical errors. For comparison, two curves corresponding to the ratios derived from ordinary concretes of 20 mm and 40 mm thickness are plotted. These indicate that both thickness of mortar specimens and γ -ray energy contribute to the γ -ray intensity attenuation. Thus, the ratio of R_γ is a powerful indicator for depth profile of chloride ion distribution in concrete.

(2) Consideration of depth information by using Time-of-Flight

Figure 14 shows a two-dimensional plot of the γ -ray spectrum and the time of flight (TOF) information from

Fig. 13 Change in intensity ratio after passing through concrete due to difference in γ -ray energy.

the time when the proton beam was emitted from the accelerator until the γ -ray was detected. The data were obtained in the measurement of the 10 mm thick mortar specimen without NaCl, and the PVDC film attached on the rear surface of the specimen (see Fig. 9). Figure 15 shows the γ -ray energy spectra gated by TOF for (a) gate below 800 μ s corresponding to the fast neutron component (shown in Fig. 2) and (b) gate above 800 μ s corresponding to the thermal neutron component (also shown in Fig. 2). Although there is no ^{35}Cl -derived γ -ray in (a), it exists in (b). This indicates that neutron capture reactions in ^{35}Cl by thermal neutrons occur near the surface of the mortar brick.

Fast neutrons are slowed down to be thermal neutrons through a thick concrete structure with a thickness of more than 200 mm. On average, this takes about several tens of microseconds, and then the thermal neutrons take part in the capture reaction. The study will be continued to confirm whether the ^{35}Cl -derived γ -rays could be observed by gating the fast neutron component in TOF when thicker (200 to 300 mm) mortar bricks without NaCl are placed in front of the mortar specimen containing NaCl.

Fig. 14 Two-dimensional plot of TOF and γ -ray energy.

Fig. 15 Gamma-ray energy spectra gated by TOF. Spectrum (a) corresponds to the gate below 800 μ s and spectrum (b) to the gate above 800 μ s.

(3) Separation using a spectral shifter

The principle of this method is the same as the separation using TOF. This is the method to obtain information on the depth direction by shifting the arrival depth of the neutron inside the specimen by placing polyethylene or other similar material with different thicknesses in front of a specimen to reduce the energy of a fast neutron. While it is necessary to consider whether TOF and the use of spectral shifters would lead to similar results, it can be thought to be effective to use instead of TOF, if the distance between the neutron source and the specimen is too short to obtain appropriate TOF separation.

5. Conclusion and future plan

The findings obtained from this study are summarized as follows:

- (1) This study for a development of a non-destructive diagnosis technique for on-site use is one of the latest achievements taking full advantage of the accelerator-driven compact neutron source, RANS, which provides thermal neutrons along with fast neutrons of a few MeV and is capable of accepting rather large samples in experimental area freely.
- (2) It is revealed that the experimental system based on NPGA with the neutron flux of 10^4 neutrons/cm²/s has adequate sensitivity for detection of the marginal chloride ion concentration of 1.2 - 2.5 kg/m³. The flux level is identified to be suitable for a portable compact neutron source.
- (3) It is proved that ^{35}Cl -derived γ -rays from a depth of 130 mm were observed. This depth is equivalent to the thickness of cover concrete.
- (4) The result of the test on the bridge sample with NPGA at RANS demonstrates quantitatively that the sample contains chloride ion concentrations between 6.1 and 12 kg/m³.

As it is clearly shown that the large background due to the neutron capture reactions on the material used in the Ge detector are observed (as shown in Fig. 5), reduction of the background is an important issue to improve the detection sensitivity. Thus optimization of the geometry concerning the incident neutron beam, the detector, and shielding structure is critical. Based on the findings obtained in these experiments, it is thought worthwhile to test various advanced methods such as multiple γ -ray intensity comparison, TOF separation and spectral shifter separation to identify the depth profile of the chloride ion distribution.

Throughout this feasibility study of NPGA application, a portable compact neutron source plays a key role for onsite use. As RANS has a weight more than 20 tons, which it is too heavy, the authors are developing a lighter weight portable neutron source, which could be less than 5 tons.

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