# STUDY ON TECHNOLOGY FOR PREVENTION AND DIAGNOSIS OF EARLY AGE FROST DAMAGE OF CONCRETE

コンクリートの初期凍害の診断および防止技術に関する研究

Jiahui Cui

Doctor of Philosophy

Division of Engineering, Course of Advanced Sustainable and Environmental Engineering

MURORAN INSTITUTE OF TECHNOLOGY

#### ABSTRACT

The most critical factors in cold weather concreting are preventing early age frost damage and guaranteeing a normal strength development. Early age frost damage is a serious problem for concrete structures in cold regions, which is caused by freezing and freeze-thaw cycles during the initial hardening stage. More specialized technologies are still needed to diagnose and prevent early age frost damage. It is required to do more research on the diagnostic and preventive approaches of early age frost damage to enhance the quality of cold weather concreting and guarantee the durability of concrete structures.

Therefore, the objective of this study was to (1) grasp the present situation of cold weather concreting in various countries and summarize the content from various guidelines by investigating Guides to Cold Weather Concreting of various countries, (2) propose effective diagnosis methods for early age frost damage, (3) investigate the effect of compressive strength development at early ages on frost resistance of concrete and (4) propose a prevention method of early age frost damage by using additive for setting time adjustment.

In chapter 2, based on the literature review of Guides to Cold Weather Concreting of various countries, it is known that most of the guidelines content in different countries' Guides to Cold Weather Concreting are similar. Different nations have different building techniques and technology, which local climates and national requirements may influence. It still needs to be understood how the minimum required compressive strength affects the frost resistance of concrete at early ages. The appropriateness of the minimum required compressive strength for cold weather concreting should be established. Meanwhile, technological development is needed to improve the diagnosis and prevention of early age frost damage.

Chapter 3 discusses the nail and pneumatic penetration test machine methods to develop accurate diagnostic methods for detecting the depth of early age frost damage. A nail penetration test can be used to determine if the concrete has been damaged by frost at an early age. However, determining the extent of early age frost damage is difficult. It is evident that the age of seven days of penetration depth has a tremendous diagnostic effect on identifying frost damage at an early age and diagnosing the damage depth. The pneumatic penetration test machine is useful for determining the extent of early age frost damage seven days after the damage occurred. With a micro-destructive degree, it can determine the depth of the damage.

In chapter 4, laboratory and outdoor exposure tests were conducted to investigate the effect of compressive strength development at early ages on the frost resistance of concrete. Air-entrained concrete that reaches a compressive strength of 5.0 MPa may effectively prevent early age frost damage and endure several freeze-thaw cycles. Frost resistance improves with the increase in compressive strength for early age concrete subjected to many freeze-thaw cycles in a hazardous water-saturated environment. For OPC concrete with a w/c of 0.5, the approximate numbers of freeze-thaw cycles needed to keep the relative dynamic modulus of elasticity over 90% for concrete with compressive strengths of 5.0, 12.0, 18.0, and 25.0 MPa were 18, 55, 90, and 124, respectively. Concrete that has reached its final setting stage may successfully withstand early age frost damage both in an air-entrained condition as well as non-air-entrained condition. Compared to laboratory tests, outdoor exposure tests showed lower frost resistance for concrete. Concrete has been reported to be less resistant to frost because of the dry-wet and freeze-thaw cycles in the natural environment.

In chapter 5, an advanced concrete finish (ACF) construction method has been developed to shorten the setting time of concrete in low-temperature environments using an additive for setting time adjustment. This chapter aims to investigate the effectiveness of the prevention method for early age frost damage using ACF additive and to recommend an appropriate program to utilize it. This study measures various tests such as slump, air content, setting time, and compressive strength. The results show that the use of ACF additive unaffected the slump and air content of fresh concrete. The setting time of concrete was significantly shortened with an increase in the amount of ACF additive, and this effect can be observed even at low temperatures. Additionally, the ACF additive can improve the early age strength within 24 hours. However, there is no difference between ACF concrete and plain concrete in compressive strength at 7 and 28 days. Adding 4 and 6 kg/m3 of ACF additive successfully prevented early age frost damage. Frozen concrete samples developed the same degree of compressive strength compared to non-frozen concrete samples after recovery curing. Therefore, 4 and 6 kg/m3 of ACF additive can be used in cold weather concreting to prevent early age frost damage.

**Keywords**: Cold weather concreting, early age frost damage, diagnosis and prevention methods, penetration test, early age of frost resistance of concrete, minimum required compressive strength, outdoor exposure test, setting time, compressive strength development.

ABSTRACT	I
TABLE OF CONTENTS	III
CHAPTER 1 INTRODUCTION	1
1.1 Background	3
1.2 Previous research	4
1.2.1 Frost damage and early age frost damage of concrete	4
1.2.2 Mechanism of early age frost damage	6
1.2.3 Prevention measures of early age frost damage	6
1.2.4 Diagnosis methods of early age frost damage	8
1.2.5 Current additive for cold weather concreting	15
1.3 Problem definition	16
1.4 Research objective and thesis organization	17
References:	20
CHAPTER 2 LITERATURE REVIEW OF GUIDES TO COLD WEATHER CO	ONCRETING OF
VARIOUS COUNTRIES	23
2.1 Overview	25
2.2 literature review of different guidelines	26
2.2 Common content in different guidelines	27
2.2.1 Application periods of cold weather concreting	27
2.2.2 Materials of concrete	
2.2.3 Mix proportion design of concrete	
2.2.4 Concrete placement temperature	
2.2.5 Preparation for cold weather concreting	
2.2.6 Concrete placement under cold weather	
2.2.7 Curing methods	

2.3 Main differences and the need for technology development for the early age frost damage
2.3.1 Minimum required compressive strength
2.3.2 Early age curing
2.3.3 Continuation curing
2.3.4 Concrete quality management at job sites
2.4 Diagnosis and prevention methods for early age frost damage in various countries43
2.4.1 Diagnosis methods for early age frost damage
2.4.2 Prevention methods for early age frost damage
2.5 Conclusion
References:
CHAPTER 3 VERIFICATION OF DIAGNOSTIC METHOD FOR EARLY AGE FROST
DAMAGE DEPTH BY PENETRATION TESTS
3.1 Overview
3.2 Experimental design
3.2.1 Penetration test by nails
3.2.2 Penetration test by pneumatic penetration test machine
<ul><li>3.2.2 Penetration test by pneumatic penetration test machine</li></ul>
3.2.2 Penetration test by pneumatic penetration test machine 50   3.3 Experimental methods 51   3.3.1 Use Materials 51
3.2.2 Penetration test by pneumatic penetration test machine503.3 Experimental methods513.3.1 Use Materials513.3.2 Mix Proportions53
3.2.2 Penetration test by pneumatic penetration test machine503.3 Experimental methods513.3.1 Use Materials513.3.2 Mix Proportions533.3.3 Mixing Method53
3.2.2 Penetration test by pneumatic penetration test machine503.3 Experimental methods513.3.1 Use Materials513.3.2 Mix Proportions533.3.3 Mixing Method533.3.4 Mold54
3.2.2 Penetration test by pneumatic penetration test machine 50   3.3 Experimental methods 51   3.3.1 Use Materials 51   3.3.2 Mix Proportions 53   3.3.3 Mixing Method 53   3.3.4 Mold 54   3.3.5 Casting 55
3.2.2 Penetration test by pneumatic penetration test machine503.3 Experimental methods513.3.1 Use Materials513.3.2 Mix Proportions533.3.3 Mixing Method533.3.4 Mold543.3.5 Casting553.3.6 Curing method and freezing method55
3.2.2 Penetration test by pneumatic penetration test machine503.3 Experimental methods513.3.1 Use Materials513.3.2 Mix Proportions533.3.3 Mixing Method533.3.4 Mold543.3.5 Casting553.3.6 Curing method and freezing method553.3.7 Test methods55

3.4.1 Penetration test by nails	
3.4.2 Penetration test by pneumatic penetration test machine	61
3.5 Conclusion	
References:	
CHAPTER 4 EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT	AT EARLY AGES
ON FROST RESISTANCE OF CONCRETE	71
4.1 Overview	73
4.2 Experimental program	75
4.2.1 laboratory tests	75
4.2.2 Outdoor exposure tests	
4.3 Experimental methods	
4.3.1 Use Materials	
4.3.2 Mix Proportions	
4.3.3 Mixing Method	
4.3.4 Mold	
4.3.5 Test methods	
4.4 Experiment result and discussion	
4.4.1 laboratory tests	
4.4.2 Outdoor exposure tests	
4.5 Conclusion	
References:	
CHAPTER 5 INVESTIGATION OF THE EFFECT OF PREVENTING EAR	RLY AGE FROST
DAMAGE USING ADDITIVE FOR SETTING TIME ADJUSTMENT	
5.1 Overview	111
5.2 Experimental design	
5.2.1 Experimental program	

5.3 Experimental methods113
5.3.1 Use Materials
5.3.2 Mix Proportions
5.3.3 Mixing Method115
5.3.4 Mold
5.3.5 Test methods
5.4 Results and discussion117
5.4.1 Fresh concrete
5.4.2 Setting time of concrete
5.4.3 Compressive strength of concrete at early ages
5.4.4 Compressive strength of concrete at 28 days
5.4.5 Prevention effectiveness of early age frost damage using ACF additive123
5.5 Conclusion131
References:
CHAPTER 6 CONCLUSIONS135
6.1 Introduction
6.2 Investigation of current situation of cold weather concreting aaccording to Guides to Cold
Weather Concreting of various countries (Chapter 2)137
6.3 Verification of diagnostic method for early age frost damage depth by penetration tests
(Chapter 3)137
6.4 Effect of compressive strength development at early ages on frost resistance of concrete
(Chapter 4)138
6.5 Investigation of the effect of preventing early age frost damage using additive for setting
time adjustment (Chapter 5)139
6.6 Summary and future work139
ACKNOWLEDGEMENTS

LIST OF PAPERS143
-------------------

## **CHAPTER 1**

## INTRODUCTION

CHAPTER 1 INTRODUCTION

#### 1.1 Background

The most critical things in cold weather concreting are preventing early age frost damage and guaranteeing a normal strength development (Architectural Institute of Japan, 2010; Architectural Institute of Japan, 2018). Early age frost damage is a serious problem for concrete structures in cold regions, which is caused by freezing and freeze-thaw cycles during the initial hardening stage. In cold weather concreting, building warm sheds, heating water and aggregates, securing a sufficient air content in the concrete, and adding anti-freeze agents were common methods to protect concrete from early age frost damage (Kim et al.,1978; Nmai, 1998; Hama and Kamada, 1997; Demirbog<sup>\*</sup>a et al., 2014). Concrete subjected to early age frost damage can cause serious problems, such as strength reduction, cracks generation, air permeability increase, and durability deterioration. To prevent the early age frost damage, the following measures have been mainly conducted in cold weather concrete strength reaches 5.0 MPa. The second is to make sure the concrete has the appropriate air content. The third is to add antifreeze to the concrete (Architectural Institute of Japan, 2010).

However, diverse countries have published their own Guides to Cold Weather Concreting for guiding construction. There are certainly differences in the requirements of guides of various countries. For example, in the guidelines of the Architectural Institute of Japan, the critical strength in the frost resistance value of early age frost damage for structure constructions is 5.0 MPa (Architectural Institute of Japan, 2010). In contrast, in the guidelines of Japan Society of Civil Engineers, concrete is commonly used in roads, bridges, and other mass concrete constructions, and the influence conditions of the water environment near the construction and the section size of concrete members are considered. The range of critical strength in frost resistance values is 5.0 to 15.0 MPa (Japan Society of Civil Engineers, 2017). It can be found that this kind of difference in the requirements of the same thing is not a rare phenomenon through reading the two standards. At present, because there is no literature on organizing the actual situation of the Guides to Cold Weather Concreting of various countries, it is hard to grasp the mainstream construction methods and technologies of cold weather concreting of various countries.

Additionally, there needs to be more research on how the minimum compressive strength needed for cold

weather concreting affects durability. Investigating how concrete's minimum essential compressive strength affects its durability is required.

On the other hand, the evaluation of the damage degree of the specimen that is subjected to early age frost damage is determined by the compressive strength recovery by curing after the early age frost damage. However, it is not straightforward to use a compressive strength test to determine the compressive strength of a concrete structure (Civil Engineering Research Institute for Cold Region, 2016). Therefore, it is necessary to test whether the concrete structure is damaged by early age frost damage with non-destructive tests and micro-destructive tests.

An increasing number of admixtures have been developed for concrete that may be used in cold weather concreting (Ramachandran, 1978; Hama, 1996; Polat et al., 2010; Wang et al., 2019; Zhang et al., 2020; Zhang et al., 2020; Alzaza et al., 2022). By using concrete admixtures, it is required to provide new methods for preventing early age frost damage. Thus, it is necessary to carry out technical development for prevention of early age frost damage of concrete.

#### **1.2 Previous research**

#### 1.2.1 Frost damage and early age frost damage of concrete

It is common practice to distinguish between "Frost damage," which occurs when concrete that has sufficiently hardened is repeatedly subjected to freeze-thaw cycles, and "Early age frost damage," which occurs when concrete that is still setting and hardening is compromised by the freeze-thaw cycles and is unable to perform as intended. Because "Frost damage" is a maintenance issue for concrete structures in terms of the durability of concrete against long-term weather effects, whereas "Early age frost damage" is more of a construction management issue for cold weather concreting, where the concrete is placed and protected to withstand the several freeze-thaw cycles that occur during construction. This is because "Early age frost damage" is a critical construction management issue for cold weather concreting (Koh and Kamada, 1978).

Except in the early stages after the concrete placement, the mechanism of early age frost damage can be treated in the same manner as long-term "Frost damage". In this case, the deterioration of concrete due to frost damage is based on the freezing and expansion of water in the microscopic voids within the concrete (Tamamushi,1961). However, in the early stages of frost damage, the concrete has not hardened sufficiently, the structure is still in the process of formation, and in extreme cases, the concrete may behave as a liquid, which requires different considerations than for concrete that has hardened sufficiently.

When water freezes to ice, its volume increases by about 9%. The pressure required to prevent this volumetric expansion can be obtained from the water state diagram (Tamamushi,1961) in Fig. 1.1. The A-D curve in the figure shows that water and ice can coexist at temperatures below 0 °C. Under the temperature and pressure to the left of this curve, the ice state is stable, while the water state is stable in the area to the right. In order to prevent water from freezing at -10°C, the pressure must be maintained on the water side of the curve, which calls for a pressure greater than 100.0 MPa.

On the other hand, the tensile strength of concrete, which varies with formulation and material, is barely 5.0 MPa and cannot contain water against such significant internal pressure. However, with some temperature drop, this pressure will, of course, exceed the tensile strength of the concrete. As a result, the water in the concrete becomes ice, and the expansion associated with this phase change causes the concrete to expand. At this stage, the concrete's elongation capacity becomes relevant. If the concrete can expand like rubber, it will not be subject to frost damage. However, the tensile elongation capacity of concrete for internal expansion is significantly less than the amount of expansion due to freezing water, resulting in damage to the concrete.



Fig. 1.1 State diagram of water

As discussed above, since concrete's tensile strength and elongation capacity are fundamentally ineffective against freezing water, mitigating the expansion caused by freezing water is an essential factor in preventing frost damage to concrete. In order for the expansion of volume not to result in significant pressure, a gas (air) must be present. Gases contract with little or no increase in pressure for a volumetric expansion of about 9%. On the other hand, for solids and liquids, the expansion pressure causes the tissue to expand. In concrete, air bubbles are the primary mitigating factor.

#### 1.2.2 Mechanism of early age frost damage

The mechanism of early age frost damage cannot be directly applied to hardened concrete. Because the dense capillary structure of the cement paste still needs to be completed. The concrete structure during setting can be described as solid aggregate and cement floating in the water. The mechanism of initial frost damage cannot be explained in a single step since there are many possible steps, from the liquid state to the presence of water in the pore space between the hardened cement and the solid aggregate. The theory of frost damage in the fresh stage of concrete is unknown. However, if the concrete is subjected to freezing at this stage, the damage is extremely severe, and removal of the damaged concrete is obviously necessary. Even when the cement has begun to set, the concrete has not yet developed strength, so it is impossible to apply theories that assume that the concrete has gained strength.

Although the effect of air bubbles on the potential for preventing deterioration was unknown, Kim et al. (1978) demonstrated that in the early stages of cement setting, the introduction of fine air bubbles by AE agents or AE reducers can be very effective, as in the case of well-cured concrete. In those investigations, freezing began at 10°C after one day of age, and curing and thawing were carried out in moist air at 20°C and 80%RH.

#### 1.2.3 Prevention measures of early age frost damage

(1) Minimum required compressive strength against early age frost damage

Koh (1959) proposed a compressive strength requirement of 5.0 MPa based on repeated experiments of freezing in air and thawing in water, which is the standard for the initial curing period required for JASS 5 cold

weather concreting. Kanda (1966) states that the loss of compressive strength in one freezing cycle is only about 5% and proposes that the initial curing period be the period during which a compressive strength of 1.5 MPa can be obtained, assuming a safety factor of 3.

#### (2) Ensuring air content

Kim et al. (1978) did experiments about Air-Entraining (AE) concrete and Non-AE concrete, made freezingthawing at the initial stage of hardening, and compared the strength after freezing and thawing with the standard increase in strength process. Fig. 1.2 shows although the AE concrete was damaged at the extremely initial stage, when the AE concrete is hardened to a particular stage, the strength is the same with the fully hardened concrete. The results showed air bubbles have a significant effect on preventing the early age frost damage due to the addition of AE agent and AE water-reducing agent.



Fig. 1.2 Effect of freezing during early age on Non-AE concrete and AE

#### (3) Accelerator for freeze protection

Accelerators for freeze protection are admixtures developed to accelerate the hydration reaction of cement and prevent initial freezing damage after concrete placement in severely cold weather. This type of admixture lowers the freezing temperature of water in concrete. It belongs to a system of admixtures that have traditionally been referred to as antifreeze because they reduce the freezing temperature of the water in concrete. It has become increasingly clear that these admixtures' initial freeze protection effect is not only due to the lowering of the freezing point but also to the acceleration of concrete hardening, and they have been called "Accelerators for freeze protection" to more accurately describe the properties of these admixtures.

Hama et al. (1996) conducted experiments on the freezing temperature, setting and hardening properties of water in the concrete, and the resistance to initial frost damage on concrete using a salt-free and alkali-free type antifreeze admixture. Experimental findings on the freezing temperature, setting, hardening characteristics, and strength-enhancing characteristics of concrete using a cold accelerator indicate that the concentration of the accelerator in concrete for practical use is about 10 vol% and the freezing temperature is about -2 to -4°C, and the difference in freezing temperature with ordinary concrete is not great. The difference in freezing temperatures is not significant. There is a limit to preventing initial frost damage by lowering the freezing temperature. However, even a melting point drop of 1-3°C under minor freezing conditions can help to significantly extend the time until the concrete freezes, suggesting a reduction in the strength correction value due to temperature (Hama et al., 1997).

When a solution of an accelerator for freeze protection, a significant amount of the unfrozen solution remains at temperatures lower than the freezing temperature and grows as sherbet-like ice. Using an accelerator for freeze protection increases the amount of water that does not freeze in concrete. When it comes to water, practically all of it freezes at 0°C. In contrast, the accelerators for freeze protection solution freezes at -12°C, 20% of the water is unfrozen at -5°C, and 10% is unfrozen at -10°C.

#### 1.2.4 Diagnosis methods of early age frost damage

According to the damage degree of the early age frost damage to concrete, the diagnostic methods of the early age frost damage can be divided into three levels. They are destructive experiments, micro-destructive experiments, and non-destructive experiments (Civil Engineering Research Institute for Cold Region, 2016). In the actual concrete construction site, we cannot use the destructive experiment to detect whether the concrete suffers from the early age frost damage. Therefore, it is essential to use the diagnosis method of the early age

frost damage of micro-destructive experiment and non-destructive experiment to diagnose the early age frost damage.

(1) Early age frost damage judgment method using torrent permeability coefficient

In recent years, non-destructive, in-situ air permeability test methods (Trent method) have attracted attention as a quality control method for concrete surface layers. Here, the failure to recover the strength of concrete that has been initially damaged by frost is not expected from the viewpoint of the concrete structure due to the loosening and coarsening of the structure, including fine cracks. It is thought that this is causing a significant increase. Therefore, Honma (2015) is examining the applicability of the air permeability test (Trent method) as a quantitative management method of the degree of damage due to the early age frost damage using concrete with a 50% water-cement ratio.

As shown in Fig. 1.3, immediately after the preparation of the specimen, the specimen was frozen at -20 ° C for 24 hours with the freezing depth changed, and then cured at 20 ° C for 28 days and sealed at 28 ° C for 28 days. The comparison of the Trent air permeability coefficient with the condition N without freezing is shown. The torrent permeability coefficient shows a more significant value as the freezing depth (exposed height) increases, reflecting the degree of damage and the frozen depth. When determining the early age frost damage, when the air permeability coefficient is based on  $10 \times 10^{-16}$  m<sup>2</sup>, the range of [10 to  $1000 \times 10^{-16}$  m<sup>2</sup>] is the influence of the early age frost damage as the criterion of the early age frost damage. And a standard of [1000 x  $10^{-16}$  m<sup>2</sup> or more] was set that had the effect of the early age frost damage.



Fig. 1.3 Relationship between Trent permeability coefficient and exposed height on the age of 28 days

Fig. 1.4 and Fig. 1.5 show the relationship between the durability index and the neutralization resistance and the Trent permeability coefficient. In the relationship between the durability index and the Trent air permeability coefficient, the durability index tended to decrease as the Trent air permeability coefficient decreased. Focusing on F-T0 (freezing start age 0h), the torrent permeability coefficient is substantial at  $1000 \times 10-16m2$ , and the durability index is significantly reduced. The torrent permeability coefficient of the other specimens F-T12 and T48 increased slightly, and the durability index decreased.

From the neutralization resistance and the torrent permeability coefficient relationship, it shows the larger neutralization rate coefficient became, the more significant torrent permeability coefficient was. It shows that a slight change in the Trent permeability coefficient affects the neutralization rate coefficient.



Fig. 1.4 Relationship between durability index and Trent permeability coefficient



Fig. 1.5 Relationship between neutralization resistance and Trent permeability coefficient

(2) Relationship between index indicating the early age frost damage level and durability

Shimakage (2016) performed an experiment on the effects of freezing temperature and freezing start age on the concrete strength. Fig. 1.6 shows the relationship between the freezing temperature and the index of the early age frost damage level. It is possible to judge that only F0 is exposed to the early age frost damage in any of the indicators of the damage level. At F0, the lower the freezing temperature, the higher the compressive strength ratio and rebound ratio, and the lower the Trent permeability coefficient, the lower the freezing temperature, and the degree of damage caused by the freezing temperature is reflected in each index. In F24 and F72, there was almost no difference in the level index of the early age frost damage depending on the freezing temperature,

which was nearly the same as that without freezing. Also, it can be seen that the lower the freezing temperature, the lower the effect of freezing temperature.



Fig. 1.6 Relationship between freezing temperature and index of the early age damage level

#### (3) Diagnosis method of the early age frost damage by visual inspection

Han (2018) experimented to determine the early age frost damage depth of concrete based on the different freezing temperatures. The specimens were made of one kind of ordinary Portland cement made in Korea, and the aggregate was made of 50% water-cement concrete using a coarse and fine aggregate of Ishiyama from Korea. To change the degree of damage caused by freezing during the initial age, the initial temperature after concrete casting was set at three levels: 20, -10 and -20 °C. Fig. 1.7 and Fig. 1.8 shows the initial frost damage depth and water absorption of the core samples collected at different initial temperatures. In the case of a healthy part without the early age frost damage and at a curing temperature of 20 °C, the water absorption rate was about 3.7%, showing a constant value. At -10 °C, it was 6.61%, and at -20 °C, it was around 7.21%, indicating that the water absorption increased from the healthy part. These results indicate that there is a clear difference in color development between the healthy part and the part that was initially damaged by frost during the drying process of the collected core specimen. At this time, the depth of the measured initial frost damage was 50 mm at -10 °C and 75 mm at -20 °C, and the depth increased as the temperature became lower.



Fig. 1.7 Cores are collected according to initial temperature.



Fig. 1.8 Water absorption of cores are collected at different initial temperatures

#### (4) Diagnosis method of the early age frost damage by drill resistance test

Yamashita (2021) proposed a method for determining the early age frost damage by using drill resistance test. In this experiment, a small vibration drill was used, with relatively small bit diameters of  $\phi 6$  mm and  $\phi 10$  mm. Fig. 1.9 shows the relationship between drilling depth and power consumption by concrete drills. Similar to the change in power consumption during core drilling, the concrete drill consumed less power in the range of 0 to 22 mm below the surface of the O-A. The concrete drill consumed less power in the range of 0 to 22 mm below the surface of the O-A. The speed of the drill slowed down when it hit the coarse aggregate, but once the drill passed through the coarse aggregate, measurements could be taken continuously. Considering the influence of aggregate, multiple drillings were conducted, and the maximum value of damage depth obtained from each result should be adopted as the estimated value of damage depth.

Since compressive strength tests are not conducted in the case of vibration drilling, this method is effective for estimating the depth of damage over a wide area and confirming that no damage has occurred in the depth direction. As with the core drilling method, measurements should be taken after 14 days of material age or after the compressive strength reaches 15.0 MPa or more. As with the core drilling method, the depth where power consumption is low should be judged as possibly damaged by comparing the relationship between the sound zone, drilling depth, and power consumption.



Fig. 1.9 Relationship between drilling depth and power consumption by concrete drills

#### (5) Observation of wet color change over time

Yamashita (2021) examined a test method by wet color. Fig. 1.10 shows the observed change in wetting color over time, with wetting color remaining in O-W and O-A.

The residual wetting color was visible at 60 minutes of aging; in O-W, the wetting color remained in the upper 20 mm area at 60 minutes of aging, and in O-A, the wetting color remained in the upper 30 mm area at 60 minutes of aging. The absence of residual wet-colored areas provides reference information that the depth of damage has not been sustained. The damage depth can be easily estimated by simply washing the cores collected during power consumption measurement during drilling, placing them indoors, and observing them. The maximum value should be used for the depth.

Sym.	0 min	30 min	60 min	90 min
I-W 91d	I-W U 4 55	I-W	I-W	I-W
I-A 91d		-A		
O-W 91d	0-W	O-W	0.W	O-W
O-A 91d		12345	0-A	0-A
Ffin- W 91d	Ffin-W	Ffin-W	Ffin-W	Ffin-W
Ffin- A 91d	Fin-A	Ffin-A	Ffin-A	Ffm-A

Fig. 1.10 Observation results of changes in wet color over time

#### 1.2.5 Current additive for cold weather concreting

In recent years, calcium silicate hydrate (C-S-H) type accelerators have been developed to improve efficiency and rationalization of production in concrete plants, which have a completely different mechanism from conventional accelerators (Koyama et al., 2015). The C-S-H type accelerators contribute to the production of secondary concrete products and are said to be effective in all temperature ranges. It may be used in cold weather concreting.

Kunizaki (2017) explored the C-S-H type accelerators in mortar and concrete. The initial strength of C-S-H type accelerators increases with C<sub>3</sub>S content because the initial hydration reaction of C<sub>3</sub>S is accelerated. Fig.

1.11 shows the setting time results of all samples at 20 °C and 5 °C. It can be found that the C-S-H type accelerators have a setting-promoting effect regardless of the cement type. With an increase in the amount utilized, the setting time is sped up even in the low-temperature environment of 5 °C.



Fig. 1.11 Setting time results of all samples at 20 °C and 5 °C

#### **1.3 Problem definition**

According to the above studies, some of the main problems of diagnosis and prevention for early age frost damage can be described as follows:

(1) Currently, it is difficult to understand the common construction techniques and technologies used in cold weather concreting across numerous nations since there needs to be literature organizing the actual state of these Guides to Cold Weather Concreting. Additionally, providing a complete prediction of the future development and trend of construction technology for cold weather concreting is challenging.

- (2) In general, the compressive strength recovery after early age frost damage is used to measure the damage degree of a specimen that has been exposed to early age frost damage. However, it is difficult to determine a concrete structure's compressive strength using a compressive strength test. Developing the non-destructive and micro-destructive test methods is required to determine if the concrete structure in the job site has been damaged by early age frost damage.
- (3) The effect of compressive strength development at early ages on the frost resistance of concrete is not clear. It is also still unknown that concrete can against how many freeze-thaw cycles with the minimum required compressive strength value. However, there is a lack of related studies on the effect of the minimum required compressive strength for cold weather concreting on durability.
- (4) With the development of admixtures for concrete, there have been more and more admixtures suitable for application in cold weather concreting. It is necessary to propose new methods to prevent early age frost damage by using concrete admixtures.

#### 1.4 Research objective and thesis organization

The objective of this study was to (1) grasp the present situation of cold weather concreting in various countries and summarize the content from various guidelines by investigating Guides to Cold Weather Concreting of various countries, (2) propose effective diagnosis methods for early age frost damage, (3) investigate the effect of compressive strength development at early ages on frost resistance of concrete and (4) propose a prevention method of early age frost damage of concrete by using additive for setting time adjustment.

This thesis includes six chapters as follows:

Chapter 1 introduces the research background on early age frost damage and related current prevention and diagnosis methods mainly used worldwide, presents the current research gaps, and lists the thesis organization.

Chapter 2 shows a literature review summary of cold weather concreting guidance of Europe, the United States, Canada, Japan, China, South Korea, and Russia. The current commonalities and differences were

compared and summarized to grasp the current situation of cold weather concreting worldwide. At the same time, the need to develop new technologies to improve the diagnosis and prevention methods for early age frost damage was identified.

Chapter 3 investigates diagnosis methods for determining the early age frost damage and detecting its depth by the penetration tests of nails and pneumatic penetration test machine. The two diagnosis methods are evaluated, and the available diagnosis method for the construction site is proposed.

Chapter 4 presents the effect of compressive strength development at early ages on frost resistance of concrete, not only laboratory but also in three years of outdoor exposure tests. The compressive strength test, hydration degree test, underwater weighing test, and freeze-thaw test were performed to discuss the influence. In addition, the applicability of minimum compressive strength for cold weather concreting is evaluated according to meteorological factors.

Chapter 5 explores the effectiveness of concrete using additive for setting time adjustment (ACF) to prevent the early age frost damage. The setting time, compressive strength of concrete with different additions are obtained and effective prevention method are proposed.

Chapter 6 shows the thesis conclusion and further research work in the future.

The following is this thesis structure:

#### **Chapter 1 Introduction**

Previous research, problem definition, research objective, thesis organization



Chapter 3 Verification of diagnostic method for early age frost damage depth by penetration tests

- Penetration test by nails
- Penetration test by pneumatic penetration test machine
- Chapter 4 Effect of compressive strength development at early ages on frost resistance of concrete
- Laboratory tests
- Outdoor exposure tests

machine

Chapter 5 Investigation of the effect of preventing early age frost damage using additive for setting time adjustment

• Influence of setting time and compressive strength development due to different addition amount

• Prevention effectiveness of early age frost damage under different addition amount

Chapter 6	Conclusions
-----------	-------------

Summary and future work

#### **References:**

Architectural Institute of Japan, 2010. Recommendation for Practice of Cold Weather Concreting, Japan.

Architectural Institute of Japan, 2018. Japanese Architectural Standard Specification JASS 5 Reinforced Concrete Work, Japan.

Möller G., Bergström S., Properties of Materials in Winter Concreting, Swedish Cement and Concrete Research Institute at the Royal Institute of Technology in Stockholm, Stockholm, Nr. 5, pp.5-79, and pp.84-100, 1962.

Meyer A., Herstellung und Nachbehandlung von Beton bei niedrigen Temperaturen, Bau-Markt, Nr, 46, 18, 3-11, Nov.1961.

Bernhardt C. J., Hardening of Concrete at Different Temperatures, RILEM Symposium Winter Concreting, Copenhagen, Feb.1956, Proc. Session B II

Scoffield H. H., Properties of Job-cured Concrete at Early Ages, ACI Disc. Vol.33, p.64, Jan.41)-Feb. 1937

McNeese D.C., EarlyFreezingofNonAir-EntrainingConcrete,J.ACI,Vol.24, pp.293-300, Dec. 1952

Cordon W. A., Freezing and Thawing of Concrete-Mechanisms and Contorol, ACI Monograph, No.3, 1966. W. A.

Demirbog`a, R., Karagöl, F., Polat, R., Kaygusuz, M.A., 2014. The effects of urea on strength gaining of fresh concrete under the cold weather conditions. Constr. Build. Mater. 64, 114-120.

Hama, Y., Kamada, E., 1997. Strength development under freezing conditions and freezing behavior of water in concrete with accerelatores for freeze protection. Concr. Res. Technol. 8 (2), 73-80. (In Japanese)

Japan Society of Civil Engineers, 2017. Standard Specifications for Concrete Structures, Materials & Construction, Japan.

Civil Engineering Research Institute for Cold Region, "Investigation of structures suspected of frost damage", 2016.1.

Ramachandran V.S., Calcium chloride in concrete — applications and ambiguities, Canad. J. Civ. Eng., 5 (2) (1978), pp. 213-221.

Hama Y., Prevention of early age frost damage by a salt-free / alkali-free antifreeze admixture, Journal of Concrete Engineering, Vol.7, No.1, pp.113-122, 1996.1.

Polat R., Demirboğa R., Karakoç M.B., Türkmen İ., The influence of lightweight aggregate on the physicomechanical properties of concrete exposed to freeze-thaw cycles, Co. Reg. Sci. Tech., 60 (2010), pp. 51-56.

Wang H., Gao X., Liu J, Influence of salt freeze-thaw cycles on the damage and the following electrical and self-sensing performance of carbon nanofibers concrete, Cold. Region. Sci. Tech., 154 (2019), pp. 95-102.

Zhang G., Yang Y.Z., Li H.M., Calcium-silicate-hydrate seeds as an accelerator for saving energy in cold weather concreting, Constr. Build. Mater., 264 (2020), p.120191.

Zhang G., Yang Y.Z., Yang H.L., Li H.M., Calcium sulphoaluminate cement used as mineral accelerator to improve the property of Portland cement at sub-zero temperature, Cem. Concr. Comp., 106 (2020), p. 103452.

Alzaza A., Ohenoja K., Dabbebi R., Illikainen, M., Enhancing the hardened properties of blended cement paste cured at 0 °C by using alkali-treated ground granulated blast furnace slag, Cem. Concr. Comp., 134 (2022), p. 104757.

Koh E., Kamada, E., Concrete frost damage and early age frost damage-about the mechanism-, Proceeding of the Japan Concrete Institute, Vol.16, No.5, 1978, pp. 1-11. (In Japanese)

Tamamushi B., Introduction to Physical Chemistry, Baifukan, p.145 (1961) (In Japanese)

Kim, M., Yokoyama, T., Tabata, M., Koh, Y., Kamada, E., 1978. An experimental study on the effect of air entrainment for frost resistance of concrete at an early age. Architectural Institute of Japan. 265, 1-10. (In Japanese)

Koh, Y., 1959. A study on winter construction of concrete in Hokkaido. Ph.D. Thesis, Hokkaido university: Hokkaido, Japan. (In Japanese)

Kanda M., Early age frost damage and its prevention, Concrete Journal, Vol.4, No.10, pp.15-20, 1966.

Hama Y., Mimori S., Kamata, E., Strength Enhancement Properties of Concrete Using Antifreeze Admixture, Proceeding of the Japan Concrete Institute, Vol.8, No.1, pp.161-170, 1997.

Honma Y., Study on determination method for early age frost damage of mortar and concrete by air permeability index and influence of early age frost damage on durability, Muroran Institute of Technology Master Thesis, 2015.

Shimakage R., Effect on freezing during early age frost damage level and durability of concrete, AIJ conference, 2016.

Han C., Effects of changes in low temperature conditions on the depth of the early age frost damage in concrete and depth determination, AIJ conference, 2018.9.

Nmai, C.K., 1988. Cold weather concreting admixtures. Cem. Concr. Compos. 20, 121-128.

Yamashita K., 2021. Mechanism of strength enhancement stagnation of concrete subjected to freezing during the setting and hardening process and measurement of the extent and depth of frost damage, Ph.D. Thesis, Muroran Institute of Technology: Hokkaido, Japan. (In Japanese)

Koyama H., Imoto H., Koizumi S., Tsuchitani T., Characteristics and Effects of Accelerator Containing C-S-H Nanoparticles, Concrete Journal, Vol.53, No.7, pp.614-621, Jul. 2015.

Kunizaki M., Study on the cold weather concreting for reducing the environmental impacts by C-S-H Type Accelerator, Muroran Institute of Technology Master Thesis, 2017.

Susuki T., Study on estimation method of concrete strength by nails penetration test, AIJ Kyushu Chapter Research Report No. 57, 2018.3.

Iwaki K., Proposal of strength control method for shotcrete by pin penetration test using air pressure, Journal of JSCE, 2000.3.

Terada K., The estimation method of concrete in structures by small size cores, Concrete Journal, 2001.

CHAPTER 1 INTRODUCTION

## **CHAPTER 2**

## LITERATURE REVIEW OF GUIDES TO COLD WEATHER CONCRETING OF

## VARIOUS COUNTRIES

#### 2.1 Overview

Since the 18th century, with the development and improvement of cement technology, concrete has become the most widely used material in engineering construction because of its advantages of high strength, durability, low cost, and applicability to diversified natural environments. Due to the influence of climate, many countries must carry out the cold weather concreting, such as the Northeast and Northwest of China, the Northeast and Hokkaido of Japan. Cold weather concreting is the process of placement, finishing, curing, and protection of concrete during cold weather, according to ACI 306 (ACI Committee 306, 2016).

However, there are several common issues for cold weather concreting in various countries, such as the early age frost damage of fresh concrete, slow strength development, and concrete quality management (ACI Committee 306, 2016, Architectural Institute of Japan, 2010) etc. Therefore, in order to guarantee the quality of cold weather concreting, diverse countries have published their own Guides to Cold Weather Concreting to solve these common issues.

In Japan, the Architectural Institute and the Society of Civil Engineers have published the cold weather concreting guidelines. However, because of the different uses of the structure constructions, the corresponding construction requirements are changed in some same issues. For example, in the guidelines of the Architectural Institute, the critical strength in the frost resistance value of early age frost damage for structure constructions is 5.0 MPa (Architectural Institute of Japan, 2010). In contrast, in the guidelines of the Society of Civil Engineers, concrete is commonly used in roads, bridges, and other mass concrete constructions, and the influence conditions of the water environment near the construction and the section size of concrete members are considered. The range of critical strength in frost resistance values is 5.0 to 15.0 MPa (Japan Society of Civil Engineers, 2017). It can be found that this kind of difference in the requirements of the same thing is not a rare phenomenon through reading the two standards. Thus, it is conceivable that there are certainly differences in the requirements of guides of various countries.

At present, because there is no literature on organizing the actual situation of the Guides to Cold Weather Concreting of various countries, it is hard to grasp the mainstream construction methods and technologies of cold weather concreting of various countries. In addition, it is also difficult to predict the development and trend of construction technology for cold weather concreting in the future comprehensively.

Therefore, the purpose of Chapter 2 is to (1) summarize the content from various guidelines; (2) grasp the current construction situation of cold weather concreting worldwide; (3) gather the information on diagnosis and prevention of early age frost damage by investigating Guides to Cold Weather Concreting of different countries.

#### 2.2 literature review of different guidelines

Table 2.1 shows the research plan of literature review. There are eight standards of cold weather concreting published by Japan (Architectural Institute of Japan, Japan Society of Civil Engineers), the United States (American Concrete Institute), Europe (RILEM), Canada (Canadian Standards Association), Russia (Non-profit partnership self-regulatory organization union of construction companies of Ural and Siberia), South Korea (Ministry of Land, Infrastructure and Transport of South Korea), and China (Ministry of Housing and Urban-Rural Development of the People's Republic of China) are selected, and most of the common contents are summarized.

Guides	Symbol	Contents
RILEM recommendations for concreting in cold		(1) Application periods of cold
weather	RILEM	
(VTT Technical Research Centre of Finland, 1988)		weather concreting;
Guida to Cold Weather Congrating		(2) Minimum required compressive
(American Concrete Institute Committee 206, 2016)	ACI	
(American Concrete Institute Committee 500, 2010)		strength;
Concrete materials and methods of concrete		(3) Materials of concrete;
construction / Test methods and standard practices for		
concrete	CSA	(4) Mix proportion design of
(Canadian Standards Association, 2014)		concrete;
Recommendation for Practice of Cold Weather		(5) Concrete placement temperature:
Concreting	AIJ	(5) Concrete placement temperature,
(Architectural Institute of Japan, 2010)		(6) Preparation for cold weather
Standard Specifications for Concrete Structures,		- concreting;
Materials & Construction	JSCE	Ċ.
(Japan Society of Civil Engineers, 2017)		(7) Concrete placement under cold

Table 2.1 Research plan of literature review
#### CHAPTER 2 LITERATURE REVIEW OF GUIDES TO COLD WEATHER CONCRETING OF VARIOUS COUNTRIES

Specification for Winter Construction of Building		weather;
Engineering	IGI	
(Ministry of Housing and Urban-Rural Development of	101	(8) Curing methods;
the People's Republic of China, 2011)		(9) Early age curing;
Recommendations for the Production of Concrete Work		
in Winter		(10) Continuation curing;
(Non-profit partnership self-regulatory organiza-tion	RNP	(11) Concrete quality management at
union of construction companies of Ural and Siberia,		
2015)		job sites.
Concrete Standard Specification		
(Ministry of Land, Infrastructure and Transport of South	MOLIT	
Korea, 2016)		

#### 2.2 Common content in different guidelines

Among the surveyed content, most of the content is similar or the same, such as the application periods of cold weather concreting, materials of concrete, concrete placement temperature, preparation for cold weather concreting, concrete placement under cold weather, curing methods, early age curing, and so on.

#### 2.2.1 Application periods of cold weather concreting

Table 2.2 shows the application periods of cold weather concreting. In the case of AIJ, the maturity factors are calculated through the following equation:

$$M_n = \sum_{z=1}^n (\theta_z + 10)$$
(1)

where  $M_n$  is the maturity factor (°D · D); z is the age (day);  $\theta_z$  is daily mean air temperature or average daily concrete temperature during z days.

The application periods of cold weather concreting of various guidelines are basically when the average daily air temperature is below 4 or 5°C based on the local meteorological conditions. When the air temperature is lower than 4 or 5°C, the fresh concrete is easy to suffer from early age frost damage. To prevent the early age frost damage, the average daily air temperature should not be lower than 4 or 5°C. Furthermore, to ensure that structure concrete can attain the design strength at 91 days after concrete placement, AIJ indicates that the maturity factor  $M_{91}$  cannot be less than 840 °D·D (Architectural Institute of Japan, 2010). To guarantee concrete quality and durability, AIJ has more strict requirements for the strength development of concrete.

Sym.	Periods
ACI	Air temperature has fallen to, or is expected to fall below, 4°C during the protection period.
AIJ	Meet either of the following conditions of 1) and 2):
	1) The average daily air temperature of ten days is less than 4°C, including the day of concrete
	placing;
	2) The period during which the maturity factor $M_{91}$ of 91 days after the concrete placing is below
	840°D·D.
	$M_n = \sum_{z=1}^n (\theta_z + 10)$
JSCE	The average daily air temperature is expected to be lower than 4 °C.
JGJ	The average daily air temperature is below 5°C for 5 days according to the local meteorological
	condition data.
RILEM	The average daily air temperature is expected to be lower than 5 °C.
CSA	The average daily air temperature is expected to be less than 5 °C.
	The average daily air temperature is expected to be lower than 5 °C, or the minimum air temperature
RNP	in one day is below 0 °C.
MOLIT	The average daily air temperature is expected to be less than 4 °C.

# 2.2.2 Materials of concrete

#### (1) Cement

Table 2.3 shows the types of cement recommended by the guides. Compared with other cement, Portland Cement, Ordinary Portland Cement, and High-early-strength Portland Cement have the advantages of high strength, high hydration heat, and good frost resistance, which are suitable for cold weather concreting. Portland

Cement and High-early-strength Portland Cement can develop high strength at the initial hardening stage, and their hydration heat is greater than that of Ordinary Portland Cement. Most guidelines recommend applying Ordinary Portland Cement and High-early-strength Portland Cement to cold weather concreting. In respect of the cement types in JGJ, Portland Cement and Ordinary Portland Cement are advised.

Besides, AIJ and JSCE indicate that it is possible to use Moderate-heat Portland Cement, Low-heat Portland Cement, and Blended Cement for cold weather concreting by taking measures such as grasping the delay in strength development in advance and ensuring the early age curing.

Sym.	Cement types
ACI	
AIJ	
JSCE	Ordinary Developed Concerns
JGJ	- Ordinary Portland Cement; - High-early-strength Portland Cement;
RILEM	
CSA	- Portland Cement.
RNP	-
MOLIT	-

Table 2.3 Types of cement recommended by the guides

#### (2) Chemical admixtures

Chemical admixtures are the ingredients in concrete other than cement, water, and aggregate that are added to the mix immediately before or during mixing, which are used primarily to improve the properties of concrete by its interfacial activity. Kim et al. (1978) reported that entraining appropriate air content effectively improves frost resistance of concrete by using the admixtures of Air-entraining and water-reducing promotion types. Thus, using the admixtures of Air-entraining and water-reducing promotion types for cold weather concreting has become a common practice, as shown in Table 2.4.

Sym.	Specifications of admixtures
ACI	Type C and Type E of ASTM C494
AIJ	
JSCE	— JIS A 6204
JGJ	GB50119
RILEM	-
CSA	Type C and Type E of ASTM C494
RNP	-
MOLIT	KSF2560

Table 2.4 Specifications of chemical admixtures in various countries

#### 2.2.3 Mix proportion design of concrete

(1) General determination method mix proportion design

The mix proportion of concrete is one of the most important factors that control the performance and quality of concrete, and mix proportion design is significant for making concrete. Because the relevant specific contents are not recorded in the guidelines of cold weather concreting in most guides, which may be recorded in other standards of their country, therefore, this chapter introduces the contents of mix proportion of concrete of AIJ and JSCE, and at the same time, the characteristics of AIJ are briefly summarized.

Fig. 2.1 shows the general determination method of mix proportion design of concrete in AIJ and JSCE. First, the maximum size of coarse aggregate, cement types, slump, and air content are selected by the structures, environmental conditions, and construction methods. Next, according to the design strength, temperatures, and other conditions to determine the strength for proportioning. After that, the water-cement ratio (w/c) is decided by considering the durability and water tightness of the structure concrete. Then, calculate the quantity of materials per unit volume such as water, cement, aggregates. Later on, carrying out the trial mixing to measure whether the properties of fresh concrete are qualified or not. Based on trial mixing results, if they are not qualified, calculate the mix proportion again until the trial mixing results are qualified. Finally, record the qualified mix

proportion of concrete. Table 2.5 lists the part of main regulations regarding the mix proportion conditions such as w/c, unit water content, slump, and air content.



Fig. 2.1 General determination method of mix proportion design of concrete

Sym.	Items	Main regulations regarding the mix proportion conditions
	w/c	Less than 65%
	Water	Less than 185 kg/m <sup>3</sup>
	Slump	Management strength of mix proportion is less than 33 N/mm <sup>2</sup> :
AIJ		below 18 cm
		Management strength of mix proportion is more than 33 N/mm <sup>2</sup> :
		below 21 cm
	Air	4.5% to 5.5%
	w/c	Less than 65%
JSCE	Water	Less than 175 kg/m <sup>3</sup>
	Slump	Determined by the types of concrete member and construction
		conditions
	Air	4% to 7%

Table 2.5 Main regulations regarding the mix proportion conditions

#### (2) Determination methods of strength for proportioning

Compressive strength is the essential performance required for concrete. Concrete strength must satisfy the design strength specified at the structural design time. The strength for proportioning is the target compressive strength considering the dispersion.

In JSCE, the strength for proportioning is the design strength multiplied by the extra coefficient. The extra coefficient under the fraction defective 5% of concrete is commonly used according to the expected strength variation coefficient. For example, assuming the strength variation coefficient is 10%, the additional coefficient is 1.2, and the strength for proportioning is the design strength multiplied by 1.2.

On the other hand, concrete used in construction (AIJ) is more complex than that used in civil engineering (JSCE). Firstly, the strength of proportioning management considering the modified strength value of concrete is calculated according to Equation (2). Then, make sure the value after adding the strength dispersion (the product of the normal and standard deviations) satisfies Eqs. 3 and 4 as the strength for proportioning. The Eqs. 2, 3, and 4 are shown below:

$$F_m = F_q + {}_{28}S_n \tag{2}$$

$$F = F_m + 1.73\sigma \tag{3}$$

$$F = 0.85F_m + 3\sigma \tag{4}$$

where  $F_m$  is the strength of proportioning management (N/mm<sup>2</sup>);  $F_q$  is quality standard strength (N/mm<sup>2</sup>);  $_{28}S_n$  is structure strength correction value (N/mm<sup>2</sup>) based on the difference between the compressive strength of the standard-cured specimen at the age of 28 days and the compressive strength of the structure concrete at the age of n days; *F* is the strength for proportioning (N/mm<sup>2</sup>) that takes the larger one of the values calculated from Eq. 3 and Eq. 4;  $\sigma$  is standard deviation of compressive strength (N/mm<sup>2</sup>).

Furthermore,  $F_m$  is required more than 24 (N/mm<sup>2</sup>). <sub>28</sub> $S_n$  is determined by either of the following methods:

- 1) Based on the expected average temperature of 28 days after concrete placement;
- 2) According to the results of maturity factor.

In the case of JSCE, the strength dispersion is determined by a fixed extra coefficient. In contrast, the

determination method of AIJ is to calculate the strength dispersion on the premise of considering the expected average temperature of 28 days after the concrete is placed and the maturity factor. And then, calculations and comparisons through the Equations are made to determine the strength for mixing proportion. The determination method of AIJ is more accurate for predicting the strength development of concrete.

## 2.2.4 Concrete placement temperature

Table 2.6 shows the concrete placement temperature of various guidelines. The contents of concrete placement temperature in JGJ and RNP are not recorded. In ACI and CSA, the minimum concrete temperature as placed is determined by the concrete section size, and the placement temperature cannot be 11°C higher than the recommended temperature. RILEM recommends keeping the concrete placement temperature from 10°C to 15°C. Due to the concrete section sizes of AIJ and JSCE being different, the temperatures are 10°C to 20°C and 5°C to 20°C, respectively. The minimum temperature of thick section size in JSCE is set to 5°C because the temperature stress caused by hydration heat easily produces cracks. In addition, MOLIT has the same regulation content as JSCE.

Sym.	Temperatures			
	Section size (mm)			
- ACI	<300	300 to 900	900 to 1800	>1800
		Minimum concrete	temperature as placed	
-	13°C	10°C	7°C	5°C
AIJ	10°C to 20°C			
JSCE		5°C t	to 20°C	
JGJ				
RILEM		10°C	to 15°C	
CSA		The regulation conte	ent is the same as ACI.	
RNP				
MOLIT		5°C t	to 20°C	

Table 2.6 Concrete placement temperature

#### 2.2.5 Preparation for cold weather concreting

The preparation works for cold weather concreting also have a significant impact on the quality of concrete. Therefore, sufficient preparation should be made before concrete placement, and the inspection work should be done according to the construction plan to ensure that the placement quality will not be affected. The primary item of preparation for cold weather concreting is to remove all snow, ice, and water from the formwork and reinforcing steel bars, as shown in Table 2.7. In addition, ACI and CSA recommend maintaining the temperature of the massive metallic embedments above -12°C (Kozikowski and McCall, 2014). AIJ requires that the temperature in the curing shed should be kept within the specified range. RILEM also states that it is essential to preheat the metal formwork and reinforcing steel bars to 3°C or 5°C.

Sym.	Items
ACI	1) Remove all snow, ice, and water from the formwork;
	2) Heat air temperature to maintain the massive metallic embedments temperature
	above -12°C.
AIJ	1) Remove all snow, ice, and water from the formwork;
	2) Confirm the temperature in the curing shed and keep that within the specified range.
JSCE	
JGJ	- Remove all snow, ice, and water from the formwork and reinforcing steel bars
RNP	- Remove an show, ice, and water from the formwork and fermoteing steel bars.
MOLIT	—
DILEM	1) Remove all snow, ice, and water from the formwork;
KILEWI	2) Recommend to preheat the metal formwork and reinforcing steel bars to $3^{\circ}$ C or $5^{\circ}$ C.
CSA	The regulation content is the same as ACI.

Table 2.7 Preparation items for cold weather concreting

#### 2.2.6 Concrete placement under cold weather

Table 2.8 shows the common attention items of concrete placement under cold weather in various guidelines. When placing concrete under cold weather, it is extremely important to take measures to deal with the external temperature related to the strength development and quality of concrete. Therefore, various guidelines emphasize this content and pay close attention to the placing process and concrete state after placing to ensure that the concrete is not frozen. At the same time, the construction units should note the meteorological conditions to prevent the severe impact of high winds and snowstorms on the concrete. Heat preservation and wind protection are crucial for the curing of concrete after placement.

Sym.	Attention items
ACI	
AIJ	1) During the placing process, the construction units should observe the uniformity
JSCE	and consistency of the concrete mixture at any time;
JGJ	- 2) The placed concrete cannot leave the exposed surface to the outside environment
	— under cold weather for a long time;
RILEM	- 3) Cover the concrete surface with a sheet or other suitable material immediately
CSA	after placement:
RNP	4) Taking measures to prevent sudden freezing from placed concrete is necessary.
MOLIT	

Table 2.8 Common attention items of concrete placement under cold weather

#### 2.2.7 Curing methods

The curing methods are displayed in Table 2.9. It mainly contains 3 types of methods: covering, thermal insulation, and heating. Generally, the curing methods are determined according to the weather conditions and section size of concrete members. For example, Concrete members with thick sections, such as foundations and slabs, can be cured by the thermal insulation method, which can use the hydration heat of the cement to maintain and increase the concrete temperature. The economy, effect, and construction conditions should be considered when selecting heating devices. It can be found that the most commonly used heating devices are gas heaters. Compared with other devices, gas heaters are simple, relatively inexpensive, and indirect heating devices that produce hot air to raise the concrete temperature.

Sym.	Methods & Materials and Equipment			
	Covering	Thermal insulation	Heating	
ACI			Gas heaters Hydronic heaters	
CSA	-		Gas heaters, riydronic heaters	
AIJ	-			
JSCE	Sheets	Thermal insulation	Gas hasters Fleetric hesters	
RNP	Curing mats	materials	Unifrared heaters	
RILEM				
MOLIT				
JGJ	-		Gas heaters, Electric heaters,	
			Infrared heaters, Steam heaters	

Table 2.9 Curing methods

# 2.3 Main differences and the need for technology development for the early age frost damage

#### 2.3.1 Minimum required compressive strength

According to the summary of different guidelines, it can be found that the technology of prevention and diagnosis of early age frost damage has always been attached importance to cold weather concreting. In addition, there are different requirements for the minimum required compressive strength in various countries. Table 2.10 shows the regulation content of minimum required compressive strength of various countries.

For protecting the concrete from the early age frost damage, it can be seen that compressive strength of 5.0 MPa is the most common minimum required compressive strength worldwide, such as in Europe, Japan, China, South Korea, and Russia. ACI recommends that concrete should reach a minimum compressive strength of 3.5 MPa before freezing. Otherwise, a single freeze-thaw cycle may cause irreparable damage. CSA recommendation is more conservative and requires that the concrete should reach a minimum compressive strength of 7.0 MPa prior to the first freeze-thaw cycle. Furthermore, from the viewpoint of ensuring the long-term durability, JSCE, ACI, CSA, and MOLIT suggest that the compressive strength values should be larger than 5.0 MPa. JSCE and MOLIT have the same regulations of the minimum compressive strength. For the concrete that exposure to severe weather conditions (multiple freeze-thaw cycles) while the surface is likely to be

saturated with water, according to the section size of the thin concrete member, ordinary concrete member, and thick concrete member, the compressive strength values are 10.0, 12.0, and 15.0 MPa, respectively. ACI proposes that the compressive strength should be more than 24.5 MPa when exposed to repeated freeze-thaw cycles while critically saturated. CSA requires that the exterior concrete flatwork needs to attain at least 32.0 MPa when exposed to freeze-thaw cycles and de-icer salts conditions, especially for the road constructions in cold regions. In the case of wet concrete continuously subjected to multiple freeze-thaw cycles, larger compressive strength values are required to ensure the long-term durability of concrete.

Therefore, the minimum required compressive strength values depend on factors such as weather conditions, size of concrete members, and exposure conditions.

Sym.	Compressive strength values
RILEM	5.0 MPa
	1) 3.5 MPa
ACI	2) When concrete exposed to repeated freeze-thaw cycles while critically saturated:
	more than 24.5 MPa
	1) 7.0 MPa
CSA	2) When exterior concrete flatwork exposed to freeze-thaw cycles and de-icer salts: at
	least 32.0 MPa
AIJ	5.0 MPa
	1) When the concrete surface is frequently saturated with water:
	depending on the *size of the cross-section
	Thin: 15.0 MPa, Ordinary: 12.0 MPa,
	Thick: 10.0 MPa
ISCE	2) When the concrete surface is rarely saturated with water:
JSCE	depending on the size of the section
	Thin: 5.0 MPa, Ordinary: 5.0 MPa,
	Thick: 5.0 MPa
	*Size of the section: Thin 20 to 30 cm,
	Ordinary 30 to 90 cm, Thick 90 to 100 cm
	Minimum air temperature > 15 °C $4.0$ MPa
JGJ	Minimum air temperature $\geq 30$ °C, 5.0 MPa
	within an temperature $\geq -50^{\circ}$ C, 5.0 Wira

Table 2.10 Regulation content of minimum required compressive strength of various countries

RNP	5.0 MPa
MOLIT	The regulation content is the same as JSCE

From this summary, it can be established that there is a positive correlation between compressive strength and frost resistance of concrete. However, the effect of compressive strength development at early ages on frost resistance of concrete is not clear. It is also still unknown that concrete can against how many freeze-thaw cycles with the minimum required compressive strength value.

#### 2.3.2 Early age curing

The aim of early age curing is to ensure the concrete strength reaches the value of minimum required compressive strength and prevent concrete from the early age frost damage after concrete placement. During this period, no part of the concrete should be frozen.

#### (1) Early age curing plan in AIJ

Since most guidelines do not document the relevant content of the early age curing plan, this Chapter briefly introduces the content of AIJ as below:

Before determining the construction plan and actual construction, the average temperature of the early age curing period is calculated according to the local meteorological data in 30 years and the current weather forecast. After that, the curing method is selected in combination with the construction conditions. When assuming the average temperature during the early age curing period, the following Eqs. 5 and 6 are used in consideration of the influence of cold waves:

$$T_{me} = T_{sme} - 4$$
 with cold waves (5)

$$T_{me} = T_{sme}$$
 without cold waves (6)

where  $T_{me}$  is the assumption value of the average temperature of the early age curing period considering the decrease in temperature;  $T_{sme}$  is the average temperature of the early age curing period within 30 years.

The determination method of curing methods is based on the  $T_{me}$ . When the  $T_{me}$  is lower than -3°C, the thermal insulation and heating methods are recommended; when the  $T_{me}$  is higher than -3°C, the covering

method is recommended. The curing temperature of the heating method, the target air temperature is higher than  $5^{\circ}$ C. In addition, no matter what method is used, it must make sure the temperature of any part of the placed concrete is higher than  $0^{\circ}$ C.

Thus, the content of the early age curing plan in AIJ is based on meteorological conditions, and the curing method is determined by assuming the average temperature during the early age curing period. This method provides effective help to ensure the quality of the early age curing.

(2) Confirmation of whether the minimum required compressive strength is reached within the early age curing period

Confirming whether the minimum required compressive strength is reached within the early age curing period commonly uses the maturity factors and field-cured cylinders of concrete. The standard method is to compute the maturity factors that achieved the critical strength in frost resistance at the same time and combine them with the compressive strength of field-cured cylinders, based on the actual compressive strength value, to determine whether the concrete attained the critical strength in frost resistance.

For example, Table 2.11 shows the results of the maturity factors (°D · D) with attained the compressive strength 5.0 MPa in AIJ. Firstly, the concrete strength can be roughly estimated according to the results recorded in Table 2.11, and then the curing time can be determined in combination with the actual construction conditions. Finally, the actual compressive strength can be confirmed according to the field-cured cylinders. If the value of critical strength in frost resistance is reached, the early age curing can be terminated. At present, most guidelines have adopted this method.

However, ACI has canceled the field-cured cylinders at the job site. The reason for cancellation is field-cured cylinders can cause confusion and unnecessary delay in construction. The use of field-cured cylinders is inappropriate and should not be allowed in cold weather concreting. This is mainly related to the difficulty in maintaining the cylinders in any approximation of the condition of the structure. In-place testing, maturity factors, or both, should be used instead. The in-place testing mainly includes non-destructive and micro-destructive tests.

Cement types	$F_m$ (MPa)								
	21	24	27	30	33	36	40	42	45
HPC*	40	35	30	25	25	25	20	20	20
OPC*	60	50	45	40	35	35	30	30	30
FA-B*	60	55	45	45	40	40	35	35	35
BFS-B*	90	80	70	60	50	45	40	40	35

Table 2.11 Results of the maturity factors (°D · D) with attained the compressive strength (5.0 MPa) in AIJ

\* HPC: High-early-strength Portland Cement, OPC: Ordinary Portland Cement, FA-B: Fly ash cement class B, BFS-B: Blast-furnace slag cement class B.

#### (3) Attention items of the end of early age curing

After confirming that the concrete strength reaches the minimum required compressive strength, the early age curing can be terminated. It should be noted that the temperature in the curing shed should be slowly reduced. Because when the temperature difference between the concrete and the external air is large, the concrete is easy to crack under the influence of cold air. Especially when heating curing is used, the concrete temperature may change rapidly when the heater is turned off, the formwork is removed, and the curing shed is removed. Therefore, to reduce the temperature difference between the concrete and the surrounding space as much as possible, these practices cannot be carried out at the same time.

#### 2.3.3 Continuation curing

The purpose of continuation curing is to cure the concrete strength to the design strength and meet the service requirements of the structure.

#### (1) Continuation curing in AIJ

Due to the lack of information on the continuation curing plan in most recommendations, the following section provides an overview of AIJ:

Since the concrete strength has reached the minimum required compressive strength, even if cold waves exist, slightly prolonging the continuation curing period ensures that concrete can get the design strength. Therefore, the assumption value of the average temperature of the continuation curing period can be estimated by Eq. (6). On the other hand, even though the curing temperature of the continuation curing period is lower than that of early age curing, it will not greatly impact the concrete strength development. In addition, it is not necessary to raise the curing temperature above 5°C.

(2) Confirmation of whether the design strength is reached within the continuation curing period

Maturity factors are combined with the compressive strength of field-cured cylinders to determine whether the concrete has reached the design strength. The concrete is evaluated to determine whether it has met the design strength based on the actual compressive strength value.

## 2.3.4 Concrete quality management at job sites

#### (1) Management of the curing temperature

The curing temperature determines the concrete strength development. Recording the concrete temperature during early age curing and continuation curing period is helpful to grasp the strength development. Fig. 2.2 shows a temperature recorder suspended from the structure. It can continuously record the concrete temperature at the measured location. It has become common to manage the curing temperature by recording the temperature.



Fig. 2.2 Temperature recorder suspended from structure

#### (2) Management of the strength development

The management of strength development is conducive to the division of early age curing and continuation curing and also improves the construction efficiency. Generally, the strength is determined by the combination of the maturity method and compressive strength test.

Over the past years, many studies have been conducted on the different methods to grasp the effect of nondestructive and micro-destructive tests on estimating concrete strength (Susuki, 2018; Iwaki, 2000; Terada, 2001). ACI recommends the non-destructive and micro-destructive tests to determine the concrete strength, as shown in Table 2.12. It indicates that the accuracy of non-destructive and micro-destructive tests is high enough.

Sym.	Methods
	Pullout strength testing,
ACI	Penetration resistance testing,
	Pulse velocity measurements
AIJ	
JSCE	
JGJ	
RILEM	Compressive strength test
CSA	
RNP	
MOLIT	

Table 2.12 Determination methods of concrete strength at job sites

Non-destructive and micro-destructive tests have replaced the compressive strength test and become the mainstream determination methods of concrete strength at job sites in America.

In addition, ACI points out that numerous commercial and proprietary computer programs have been developed that generally employ the finite element or finite difference models changing boundary and initial conditions. These are useful to predict not only temperature but, combined with the maturity concept, to predict the strength of the concrete at later ages.

# 2.4 Diagnosis and prevention methods for early age frost damage in various countries

#### 2.4.1 Diagnosis methods for early age frost damage

Through the investigation of guideline content, it can be found that the current methods for diagnosing early age frost damage in various countries are mainly the compressive strength test or the concrete surface rebound hammer test.

Generally, the evaluation of the damage degree of the specimen that is subjected to early age frost damage is determined by the compressive strength recovery by curing after the early age frost damage. However, ACI points out that using compressive strength tests for concrete strength confirmation at construction sites also can cause a number of mismanagement factors that affect the accuracy of the tests.

ACI proposes that non-destructive tests or micro-destructive tests at construction sites should be used. In addition, other countries also mentioned that diagnosis methods of early age frost damage should be simple and convenient. It is necessary to test whether the concrete structure is damaged by early age frost damage with nondestructive or micro-destructive tests.

#### 2.4.2 Prevention methods for early age frost damage

At present, the methods of preventing early age frost damage in various countries are mainly carried out from the following three aspects: firstly, it is recommended that the placed concrete is cured at normal temperature until the concrete strength reaches 5.0 MPa; secondly, it is to make sure the concrete has the appropriate air content; thirdly, it is to add anti-freeze additive to the concrete for preventing the early age frost damage.

However, with the deepening of research on early age frost damage and the development of concrete additives, technical methods to prevent early age frost damage should also be developed.

### 2.5 Conclusion

According to the contents and summaries in this chapter, the conclusions are given as follows:

(1) Most of the regulations of the Guides to Cold Weather Concreting of various countries are relatively similar, such as materials of concrete, curing methods, and preparation for cold weather concreting.

(2) Each country has their unique construction methods and technologies, which may be related to various countries' climatic conditions and national standards.

(3) The effect of the minimum required compressive strength values at early ages on frost resistance is still

unclear. It is necessary to determine the applicability of minimum compressive strength for cold weather concreting.

(4) With the development of new technologies, the diagnosis methods and prevention methods for early age

frost damage also should be developed.

#### **References:**

American Concrete Institute Committee 306-66, 1966. Recommended Practice for Cold Weather Concreting, America.

American Concrete Institute Committee 306, 2016. Guide to Cold Weather Concreting, America.

Architectural Institute of Japan, 2010. Recommendation for Practice of Cold Weather Concreting, Japan.

Japan Society of Civil Engineers, 2017. Standard Specifications for Concrete Structures, Materials & Construction, Japan.

Ministry of Housing and Urban-Rural Development of the People's Republic of China, 2011. Specification for Winter Construction of Building Engineering, China.

Ministry of Land, Infrastructure and Transport of South Korea, 2016. Concrete Standard Specification, South Korea.

RILEM recommendations for concreting in cold weather; VTT Technical Research Centre of Finland: Espoo, Finland, 1988.

Recommendations for the Production of Concrete Work in Winter; Non-profit partnership self-regulatory organization union of construction companies of Ural and Siberia: Chelyabinsk, Russia, 2015. (In Russian)

Concrete Materials and Methods of Concrete Construction/Test Methods and Standard Practices for Concrete; Canadian Stand-ards Association: Toronto, ON, Canada, 2014.

Kim, M., Yokoyama, T., Tabata, M., Koh, Y., Kamada, E., 1978. An experimental study on the effect of air entrainment for frost resistance of concrete at an early age. Architectural Institute of Japan. 265, 1-10. (In Japanese)

Powers, T.C. Prevention of frost damage to green concrete. RILEM Bull. 1962, 14, 120–124.

Koh E., Kamada, E., Concrete frost damage and early age frost damage-about the mechanism-, Proceeding of the Japan Concrete Institute, Vol.16, No.5, 1978, pp. 1-11. (In Japanese)

ASTM C494, "Standard Specification for Chemical Admixtures for Concrete", ACI Committee E-701, 2013.

JIS A 6204, "Chemical admixtures for concrete", Japanese Industrial Standard Committee, 2006.

GB50119, "Code for concrete admixture application", Ministry of Housing and Urban-Rural Development of the People's Republic of China, JGJ, 2013.

Kozikowski, R. L., McCall, W. C., Supernant, B., "A., "How Does a Cold Reinforcing Bar Affect Fresh Concrete?" Concrete International, V. 36, No. 4, Apr., pp.49-56, 2014.

Susuki, T., Study on estimation method of concrete strength by nail penetration test, AIJ Kyushu Chapter Research Report No. 57, 2018.

Iwaki, K., Proposal of strength control method for shotcrete by pin penetration test using air pressure, Journal of JSCE, 2000.

Terada, K., The estimation method of concrete in structures by small size cores, Concrete Journal, 2001.

# **CHAPTER 3**

# VERIFICATION OF DIAGNOSTIC METHOD FOR EARLY AGE FROST DAMAGE

# **DEPTH BY PENETRATION TESTS**

#### 3.1 Overview

To investigate the early age frost damage depth of concrete on the construction site. It is necessary to test whether the concrete structure is damaged by early age frost damage with non-destructive tests and microdestructive tests. Several studies have been conducted on the different methods to grasp the depth of the early age frost damage. However, in these studies, it has not yet been able to accurately detect the depth of the early age frost damage with non-destructive or micro-destructive tests.

On the other hand, it is helpful to test the depth of early age frost damage by referring to the estimation methods of compressive strength of concrete studies, which are micro-destructive tests.

In this study, we tried to use the penetration tests to detect the depth of the early age frost damage with the micro-destructive degree.

#### 3.2 Experimental design

#### 3.2.1 Penetration test by nails

Table 3.1 shows the experimental plan. The mortar specimens were prepared using ordinary Portland cement (OPC) and 50% water to cement ratio. All specimens were poured as  $\varphi 100 \times 200$  mm cylinder sizes.

N was placed at a 20°C 60 %RH room after casting, and it was sealed curing continued until the age of 7 days. When the early age frost damage is imparted to F, it was frozen at -20°C 12 hours using a refrigerator, and then placed at a 20°C 60% room, and it was sealed curing continued until the age of 28 days. Fig. 3. 1 shows the exposure height that is set to 0mm, 20mm, 40mm, 60mm. It changed the depth of early age frost damage. The freezing period is not included in the age.

Same hal	Comont w/c		Exposure	Freezing	conditions	Curing after freezing	
Symbol	Cement	[%]	[mm]	Temp	Time	Tomm [9C]	Time
				[°C]	[h]	Temp [*C]	[d]
Ν			_	Non-fr	reezing		
F-0			0				
F-20	OPC	50	20	-20	12	20	7
F-40			40				
F-60			60				

Table 3.1 Experimental plan



Fig. 3.1 Outline Fig.

# 3.2.2 Penetration test by pneumatic penetration test machine

Table 3.2 shows the experimental plan. The mortar specimens were prepared using ordinary Portland cement (OPC) and 50% water to cement ratio. According to the requirement of the pneumatic penetration test method, the specimen size was poured as 308× 228×152 mm.

N was placed at a 20 ° C 60 %RH room after casting, and it was sealed curing continued until the age of 3, 7, 14, 28 days. When the early age frost damage is imparted to F, it was frozen at -20 ° C 12 hours using a refrigerator, and then placed at a 20 ° C 60% room, and it was sealed curing continued until the age of 28 days. The exposure height condition is the same as Fig. 3.1 that is set to 0mm, 20mm, 40mm, 60mm. It changed the depth of early age frost damage. The freezing period is not included in the age.

Same hal	Coment w/c		Exposure	Freezing	conditions	Curing after freezing		
Symbol	Cement	[%]	[mm]	[%]	Temp	Time	Temp [%]	Time
				[°C]	[h]	iemp [ C]	[d]	
Ν				Non-freezing			2	
F-0		50	0			20	3	
F-20	OPC		20	-20	12		/	
F-40			40				14	
F-60			60				20	

Table 3.2 Experimental plan

# 3.3 Experimental methods

# 3.3.1 Use Materials

(1) Ordinary Portland Cement

		Ordinary Portland Cement			
		JIS Standard Value	Experimental Results		
Der	nsity (g/cm <sup>3</sup> )	-	3.17		
Specific S	urface Area (cm <sup>3</sup> /g)	More than 2500	3500		
	Water (%)	-	28.0		
Setting	Initial Setting (h $\cdot$ mm)	More than 60 min	1-45		
	Final Setting (h $\cdot$ mm)	Less than 10 h	3-00		
Soundness	Soundness Pat Test		Qualified		
	1d	-	-		
Compressive	3d	12.5	32.9		
Strength	7d	22.5	51.0		
$(N/mm^2)$	28d	42.5	65.8		
	91d	-	-		
Heat evolution	7d	-	332		
(J/g)	28d	-	387		
	Magnesium Oxide	Less than 5.0	2.1		
Chemical	Sulfur Trioxide	Less than 3.5	1.9		
Composition (%)	Sulfur Dioxide	-	21.4		

Table 3.3 Chemical composition and physical properties of OPC

CHAPTER 3 VERIFICATION OF DIAGNOSTIC METHOD FOR EARLY AGE FROST DAMAGE DEPTH BY PENETRATION TESTS

	Aluminum Oxide	-	5.5
	Ferric Oxide	-	2.8
	Calcium Oxide	-	64.3
	Ignition Loss	Less than 5.0	0.56
	Total Alkali	Less than 0.75	0.25
	Chloride Ion	Less than 0.035	0.012
	Tricalcium Silicate	-	52
Minoral	Dicalcium Silicate	-	23
composition (%)	Tricalcium Aluminate	-	10
	Tetracalcium		0
	Aluminoferrite	-	7

Ordinary Portland Cement is the basic ingredient of concrete. It binds coarse aggregate with fine aggregate. Cement is manufactured through a closely controlled chemical combination of calcium, silicon, aluminum, iron and other ingredients. In this study, OPC was used as compliance with JIS standard. Table 3.3 shows Chemical composition and physical properties of OPC.

#### (2) Fine aggregate

In this study, we used the Noboribetsu land sand. Table 3.4 shows the physical test results by the JIS standard test method. Table 3.5 shows the sieving test results. Fig. 3.2 shows the particle size distribution.

Density	(g/cm <sup>3</sup> )	Water	Mass of unit	Solid volume
Surface dry	Absolute dry	absorption [%]	volume [kg/ℓ]	percentage [%]
2.57 2.49		2.98	1.63	65.6

Table 3.4 The physical test results

Table 3.5 The sieving test results

Sample aggregate		Percer	ntage of	mass pa	ssing thro	ough sieve	
Nominal size of sieve [mm]	0.15	0.3	0.6	1.2	2.5	5	10
coarse aggregate	95	80	63	33	8	0	0



#### (3) Water

In this study, we used the tap water of our university.

#### 3.3.2 Mix Proportions

Table 3.6 shows the preparation table.

Table 3.6 Mix Proportions

w/c	C.S	Unit mass[kg/m <sup>3</sup> ]					
[%]	C:8	Water	Cement	Fine aggregate			
50	1:3	258	517	1550			

### 3.3.3 Mixing Method

The fine aggregate was made surface dry on the day before the mortar was made, and the condition of the surface dry state was checked and adjusted on the day before use. The mixing was based on JIS R 5201-1997 "Physical test method of cement". A mixer named LC-607A (nominal capacity: 60  $\ell$ , rotation speed: 48 rpm, motor output: 2.2 kW, manufactured by Sanyo Testing Machine Co., Ltd.) was used for mixing as shown in Fig. 3.3. Firstly, putting OPC and fine aggregate in mixer  $\rightarrow$  mixing for 30 seconds  $\rightarrow$  using a spoon to scrap for 1 minute  $\rightarrow$  mixing for 2 minutes. Immediately after

mixing, carefully pour the mortar into the container.



Fig. 3.3 Mixer

# 3.3.4 Mold

The mold  $\phi 100 \times 200$  (mm) cylinder-type plastic was used in the penetration test by nails to determine the early age frost damage. Fig. 3.4 shows the mold.

The mold  $308 \times 228 \times 152$  (mm) container was used in the penetration test by pneumatic penetration test machine to determine the early age frost damage. Fig. 3.5 shows the mold.



Fig. 3.4  $\varphi$ 100×200(mm) cylinder-type plastic mold



Fig. 3.5 308× 228×152 (mm) container

#### 3.3.5 Casting

The casting method is to divide the cylindrical mold into two layers, pierce each segment 15 times with a ram, and hit with a mallet. After that, the top of the mold was covered with a vinyl sheet to prevent moisture from escaping.

#### 3.3.6 Curing method and freezing method

N was placed at a 20 °C 60 %RH room after casting, and it was sealed curing continued until the age of 28 days. When the early age frost damage is imparted to F, it was frozen at -20 °C 12 hours using a refrigerator, and then placed at a 20 °C 60 % RH room, and it was sealed curing continued until the age of 28 days. Fig. 3.1 shows the exposure height that is set to 0 mm, 20 mm, 40 mm, 60 mm. It changed the depth of early age frost damage. The freezing period is not included in the age.

#### 3.3.7 Test methods

#### 3.3.7.1 Fresh properties

The test is performed by JIS A 1128 "Test method for air volume of fresh concrete by pressure (air chamber pressure method)". The specimen is poured in three layers, approximately 1/3 of the container. Each layer is pierced 15 times by a stick. The outside of the container is hit with a mallet about 15 times.

#### (2) Flow

The flow test is performed according to JIS R 5201 "Physical test method for cement". By using a flow table, a flow cone, and a stick, mortar is packed into two layers, each half of the flow cone and the mortar is struck 15 times with a stick, and then falls into the mortar once a second. 15 times and the diameter after the mortar spreads is measured as the flow value. Read the scale up to 1 mm.

#### 3.3.7.2 Rebound degree method

<sup>(1)</sup> Air content

Fig. 3.6 shows the shows the N-type rebound hammer used in the experiment. In this experiment, the rebound was measured using a rebound hammer according to JIS A1155 "Method of measuring the rebound of concrete". The arithmetic average of all measured values is taken as the measured hardness at that location. If the error is about 20% or more of the average value, the average value is calculated by discarding the error and supplementing the alternative. The estimated strength is calculated by the following equation.

$$F_c = 0.73R + 10$$

where,  $F_c$  is the estimated strength (N / mm<sup>2</sup>); R is the measured hardness.



Fig. 3.6 N-type rebound hammer

#### 3.3.7.3 Penetration test by nails

Using a tool, as shown in Fig. 3.7, a 1.4kg weight was fell freely from a height of 860mm and hit to a nail. It was supported by a PVC pipe to assist the tool. A 15mm diameter, a thickness of about 10mm sponge with a hole in the center, was attached to the specimen using an instant adhesive to prevent the nail from jumping. The penetration test was performed with the nails inserted in the holes. The nail is a commercial concrete nail. The nail is total length of 38 mm and thickness of  $2.8 \pm 0.1$  mm as shown in Fig. 3.8. Each time of the penetration test by nails, the nail penetration amount was measured from the exposed length of the nail using an electronic caliper. The four-measure points were performed on the upper surface of the specimen. If the nail becomes bend in the middle, the test will do one more time again. Fig. 3.9 shows the condition of nails fixed on the specimen. Fig. 3.10 shows the condition of after the nails are hit into the specimen.

# CHAPTER 3 VERIFICATION OF DIAGNOSTIC METHOD FOR EARLY AGE FROST DAMAGE DEPTH BY PENETRATION TESTS



Fig. 3.7 PVC pipe and weight

Fig. 3.8 Commercial concrete nail



Fig. 3.9 The condition of nails fixed on the specimen



Fig. 3.10 The condition of after the nails are hit into the specimen

# 3.3.7.3 Penetration test by pneumatic penetration test machine

This is a test machine, as shown in Fig. 3.11, that drives a dedicated pin into concrete by air pressure. It can measure the penetration depth and estimate the concrete strength.

It is easy to use. Air pressure can obtain stable pressure. There is no restriction on penetration direction.

First, connect the parts of the machine as shown in Fig. 3.12. Then, open the air pressure valve and adjust the

air pressure to 1.6Mpa. Before the experiment, to ensure the stability of pressure, the anvil was first used for testing, as shown in Fig. 3.13. The depth of penetration is measured with an electronic vernier caliper.

To avoid cracks which is caused by pneumatic pin hit, it is necessary to make sure the distance between two measuring points is more than 70mm. Each specimen was set to do the penetration test at 6 measuring points on the upper. And the pneumatic power is set to 1.6MPa. The penetration amount of each time is recorded, and the average value is calculated.



Fig. 3.11 Pneumatic penetration test machine



Fig. 3.12 Machine connection



Fig. 3.13 Pressure testing by anvil

# 3.4 Results and discussion

### 3.4.1 Penetration test by nails

(1) Rebound degree method

Fig. 3.14 shows the rebound degree on the age 7days. It is seen that F0, F20, F40, and F60 have a lower rebound degree compared to N. The early age frost damage of all F specimens can be determined by rebound degree.



Fig. 3.14 Rebound degree on the age 7 days

Fig. 3.15 shows the rebound degree ratio based on N of the age of 7 days. The rebound degree ratio was smaller than N. It can be confirmed that all the F specimens were subjected to the early age frost damage. There was a noticeable difference between N and F, but the difference is small in F.



#### (2) Penetration test by nails

Fig. 3.16 shows the relationship between times of hit and average nail penetration depthy. There is a difference in penetration depth between N and F0, F20, F40, F60, and the difference becomes obvious as the times of hit increases. The difference in penetration depth between F0, F20, F40 and F60 is small. It can be considered that the average nail penetration amount is possible to determine the early age frost damage. But it is difficult to detect the damage depth due to the penetration depth is much smaller than the freezing exposure height.



Fig. 3.17 shows the relationship between rebound degree and the nail penetration depth. Compared with N, the rebound degree of F0, F20, F40, and F60 is smaller, and the penetration amount is larger. However, the distribution of F is very discrete and cannot present a linear relationship. From the penetration depth of F, it is only about 5mm, so it is difficult to calculate the depth of the early age frost damage.



Fig. 3.17 Relationship between rebound degree and nail penetration depth

From the above results, it is possible to diagnose whether the concrete is subjected to early age frost damage by a nail penetration test. But it is difficult to diagnose the depth of early age frost damage. It can be considered that the hardness of the nails does not reach the level where it can fully penetrate the specimens, and the position where the weight free falls to hit the nail also affected the penetration depth.

#### 3.4.2 Penetration test by pneumatic penetration test machine

#### (1) Rebound degree method

Figs. 3.18, 3.19, 3.20, and 3.21 show the the rebound degree on the age of 3, 7, 14, and 28 days. It is seen that F0, F20, F40, and F60 have a lower rebound degree compared to N. The early age frost damage of all F specimens can be determined by rebound degree.

VERIFICATION OF DIAGNOSTIC METHOD FOR EARLY AGE FROST DAMAGE DEPTH BY PENETRATION TESTS









Fig. 3.20 The rebound degree on the age of 14 days
CHAPTER 3

VERIFICATION OF DIAGNOSTIC METHOD FOR EARLY AGE FROST DAMAGE DEPTH BY PENETRATION TESTS



Fig. 3.21 The rebound degree on the age of 28 days

From the above results, the N rebound degree of the age of 28 days is larger than another ages.

Figs. 3.22, 3.23, 3.24, and 3.25 show the rebound degree ratio based on N of the age of 3, 7, 14, and 28 days. The rebound degree ratio was smaller than N. It can be confirmed that all the F specimens were subjected to the early age frost damage. There was a noticeable difference between N and F, but the difference is small in F.



Fig. 3.22 Rebound degree ratio based on N at 3 days



Fig. 3.25 Rebound degree ratio based on N at 28 days

<sup>(2)</sup> Penetration test by pneumatic penetration test machine

Figs. 3.26, 3.27, 3.28, and 3.29 show the average pin penetration depth on the age of 3, 7, 14, and 28 days. It can be seen that the penetration depth at 3days is larger than another ages. It can be considered that the average nail penetration depth is possible to determine the early age frost damage. It is confirmed that the penetration amount increased as the early age frost damage depth increased.



Fig. 3.26 Average pin penetration depth on the age of 3 days



Fig. 3.27 Average pin penetration depth on the age of 7 days



Fig. 3.28 Average pin penetration depth on the age of 14 days



Fig. 3.29 Average pin penetration depth on the age of 28 days

Figs. 3.30, 3.31, 3.32, and 3.33 shows the relationship between early age frost damage depth and pin penetration depth on the age of 3, 7, 14, and 28 days. On the age of 3, 14, and 28 days, they are low correlated functional relationships between pin penetration depth and early age frost damage depth. The damage depth is difficult to calculate by using the penetration depth of the age 3, 14, and 28 days. On the age 7 days, there is a highly correlated functional relationship between pin penetration depth and early age frost damage depth, and the depth can be roughly calculated by using penetration amount. This function relation is very approximate and can be used for reference.



Fig. 3.30 Relationship between early age frost damage depth and pin penetration depth (3 days)



Fig. 3.31 Relationship between early age frost damage depth and pin penetration depth (7 days)



Fig. 3.32 Relationship between early age frost damage depth and pin penetration depth (14 days)



Fig. 3.33 Relationship between early age frost damage depth and pin penetration depth (28 days)

From the above results, it can be seen that the penetration amount of the age 7 days has an excellent diagnosis effect for determining the early age frost damage and detecting the damage depth. The penetration test by pneumatic penetration test machine is effective to diagnose the depth of early age frost damage on the age 7 days. It can roughly detect the damage depth with a micro-destructive degree.

# 3.5 Conclusion

In this chapter, the study on verification of diagnostic method for early age frost damage depth by penetration tests is carried out. It was explored about the relationship between the early age frost damage depth and penetration amount. The penetration test by nails and pneumatic penetration test machine were examined.

From all the results, the following summaries can be drawn:

(1) It is possible to diagnose whether the concrete is subjected to early age frost damage by a nail penetration test. But it is difficult to diagnose the depth of early age frost damage.

(2) It can be seen that the penetration amount of the age 7 days has an excellent diagnosis effect for determining the early age frost damage and detecting the damage depth.

(3) The penetration test by pneumatic penetration test machine is effective to diagnose the depth of early age frost damage on the age 7 days. It can roughly detect the damage depth with a micro- destructive degree.

# **References:**

Hama Y., Mimori S., Kamata, E., Strength Enhancement Properties of Concrete Using Antifreeze Admixture, Proceeding of the Japan Concrete Institute, Vol.8, No.1, pp.161-170, 1997.

Honma Y., Study on determination method for early age frost damage of mortar and concrete by air permeability index and influence of early age frost damage on durability, Muroran Institute of Technology Master Thesis, 2015.

Shimakage R., Effect on freezing during early age frost damage level and durability of concrete, AIJ conference, 2016.

Han C., Effects of changes in low temperature conditions on the depth of the early age frost damage in concrete and depth determination, AIJ conference, 2018.9.

Nmai, C.K., 1988. Cold weather concreting admixtures. Cem. Concr. Compos. 20, 121-128.

Yamashita K., 2021. Mechanism of strength enhancement stagnation of concrete subjected to freezing during the setting and hardening process and measurement of the extent and depth of frost damage, Ph.D. Thesis, Muroran Institute of Technology: Hokkaido, Japan. (In Japanese)

Koyama H., Imoto H., Koizumi S., Tsuchitani T., Characteristics and Effects of Accelerator Containing C-S-H Nanoparticles, Concrete Journal, Vol.53, No.7, pp.614-621, Jul. 2015.

Kunizaki M., Study on the cold weather concreting for reducing the environmental impacts by C-S-H Type Accelerator, Muroran Institute of Technology Master Thesis, 2017.

# **CHAPTER 4**

# EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT AT EARLY AGES ON FROST RESISTANCE OF CONCRETE

#### 4.1 Overview

The minimum required compressive strength is a reference value defined from the experimental results. Previous studies (Koh, 1959; Powers, 1962; American Concrete Institute Committee 306-66, 1966; RILEM, 1963; Voellmy, 1956; Rastrup, 1964) have been conducted to determine the minimum required compressive strength values. Koh (1959) proposed that 5.0 MPa should be taken for the minimum required compressive strength value, which was obtained from repeated experiments of freeze-thaw cycles. Powers (1962) reported that the hydration process consumes the water in fresh concrete during strength development, and also calculated the degree of dryness at which frost damage will not occur. Moreover, compressive strength of 2.9 MPa was obtained as the limit value. Subsequently, American Concrete Institute Committee 306-66 (1966) specified 3.5 MPa as a protective compressive strength value for concrete against once freezing. RILEM guideline (1963) for cold weather concreting stated that compressive strength of at least 5.0 MPa can resist one to several freezethaw cycles when there is no external water supply considering different water to cement ratios, curing temperatures, and cement types. Voellmy (1956) mentioned that if the concrete surface is often saturated with water, compressive strength of 15.0 MPa is required to resist frost damage. Rastrup (1964) also introduced the theories and calculations on the minimum required compressive strength. With decades of practice in actual cold weather concreting, it is widely recognized that concrete with the minimum required compressive strength can resist early age frost damage.

However, according to recent studies, even though concrete has reached a compressive strength of 5.0 MPa, freezing and multiple freeze-thaw cycles still have adverse effects on the durability of concrete. Koh et al. (2013) verified the pertinence of the minimum compressive strength of 5.0 MPa. Concrete specimens were subjected to 30 freeze-thaw cycles between +4°C and -18°C in a laboratory test, which is approximately equivalent to freeze-thaw actions that would occur within one year in Korea's real, natural environment. And then, the frozen concrete was performed with 28d 20°C standard curing. The results showed no difference in compressive strength between frozen concrete and non-frozen concrete. However, frost resistance and chloride ion penetration resistance have decreased significantly. Similarly, Choi et al. (2017) indicated that the concrete,

which exhibited a compressive strength exceeding 5.0 MPa, suffered from freezing at -20°C for 24 h, the compressive strength values of frozen concrete and non-frozen concrete were approximately the same, while the frost resistance of frozen concrete was obviously lower than that of non-frozen concrete. It can be found that 5.0 MPa as the minimum required compressive strength to prevent early age frost damage is reasonable in strength development. Nevertheless, once concrete at an early age is subjected to severe freezing or multiple freeze-thaw cycles that exceed the protective capacity of minimum required compressive strength, the durability of concrete will be damaged.

According to the Guide to Cold Weather Concreting (American Concrete Institute Committee 306, 2016), the cold weather concreting application period is when the air temperature has fallen to or is expected to fall below 4°C during the protection period. It means that the period generally extends from winter to spring. During the period before spring, even if concrete reached the minimum required compressive strength, its durability is also possible to be deteriorated due to sudden cold waves, repeated freeze-thaw cycles, and insufficient early age curing based on the research findings of Koh et al. (2013) and Choi et al. (2017). At present, however, the related studies about the minimum required compressive strength for cold weather concreting on durability are deficient. Hence, it is necessary to investigate the effect of the minimum required compressive strength of concrete on its durability.

Generally, in experimental conditions, evaluating the frost resistance of concrete is mainly for completely hardened concrete. The test method for frost resistance of concrete is to place concrete into temperatureadjustable equipment and subject the concrete to 300 freeze-thaw cycles, such as ASTM C 666 (American Society for Testing and Materials International, 2015), JIS A 1148 (Japan Industrial Standards Committee, 2010), and GB/T50082-2009 (National Standard of the People's Republic of China, 2009). Suppose the concrete with the minimum compressive strength is subjected to 300 freeze-thaw cycles based on ASTM C 666 (American Society for Testing and Materials International, 2015). In that case, it is possible to determine the deterioration behaviors of concrete with the minimum compressive strength within 300 freeze-thaw cycles. If the frost resistance of concrete with the minimum compressive strength can be grasped, it will help prevent the early age frost damage. Besides, to connect with concrete structures in the natural environment, the frost damage deterioration behavior of concrete at early ages can be estimated by meteorological factors. Estimating frost damage deterioration behavior of concrete based on meteorological factors has already made much progress (Hasegawa and Hon, 1979; Hama et al., 1999; Quy et al., 2020). Hama et al. (1999) proposed an estimation method about the ASTM (American Society for Testing and Materials) equivalent number of cycles ( $Cy_{ASTM-sp}$ ) based on meteorological factors. It can convert the freeze-thaw actions received per year from the temperature and environmental conditions into several freezing and thawing cycles in the standard of ASTM C 666 A method (American Society for Testing and Materials International, 2015) to determine the regional characteristics of frost damage of cement-based materials. It is well known that the early age frost damage always occurs after construction from winter to spring. Concrete structures exposed to the natural environment often experience freeze-thaw actions during this period. It is possible to estimate the theoretical number of freeze-thaw cycles of concrete structures experienced in cold regions during this period according to the estimation method of  $Cy_{ASTM-sp}$ .

This chapter is divided into two parts to investigate the effect of compressive strength development at early ages on the frost resistance of concrete according to laboratory tests and 3 years outdoor exposure tests.

#### 4.2 Experimental program

#### 4.2.1 laboratory tests

Table 4.1 shows the experimental design of the laboratory tests. The symbol of the N specimen means the non-frozen concrete as a reference specimen for Series 1 and 2. The symbol of the F and S represent the frozen concrete and required compressive strength value. For example, F-S5 means the frozen concrete with 5.0 MPa. Especially, the specimen of F-T12 in Series 1 was the frozen concrete with a pre-curing time of 12 h. The pre-curing time before freezing of each specimen was determined by the pre-experiment. Experimental items in the laboratory tests were compressive strength, hydration degree, total porosity, and freezing and thawing resistance, as shown in Table 4.1.

**CHAPTER 4** 

In this study, the main experiments were divided into two series due to different investigation objectives. Fig.4.1 shows the flowchart of the main experiments. The concrete mixture was manufactured, and then it was cast into  $\emptyset 100 \times 200$  mm cylindrical plastic molds and  $75 \times 75 \times 400$  mm steel molds. Afterwards, the specimens were laid in a room at 20°C and 60% relative humidity. For the reference specimen, after curing for 28 days, the freeze-thaw test was carried out until 300 cycles according to JIS A 1148 A method (Japan Industrial Standards Committee, 2010).

In Series 1, it contained the case of early age frost damage of concrete (F-T12) and the conditions of concrete with different compressive strength values (F-S5, F-S12, F-S18, and F-S25), which is aimed at evaluating the physical recovery performance of concrete that was subjected to several freeze-thaw cycles at early ages. Based on the pre-experiment results, the compressive strength values of the frozen specimens in Series 1 were determined by the compressive strength test after curing for 12 h, 24 h, 44 h, 50 h, and 75 h, respectively. After confirming the compressive strength, the specimens of Series 1 were transferred to an adjustable temperature chamber for freeze-thaw cycles in air condition for 3 cycles, and one cycle was 12 h -20 °C and 12 h +5 °C, which was used to simulate the daytime and nighttime temperatures in the Hokkaido of Japan during winter. After 3 freeze-thaw cycles, the specimens were replaced in a room at 20 °C and 60 % relative humidity to perform the recovery curing until 31 days. Experimental items were performed, and the freeze-thaw test was conducted until 300 cycles in accordance with the JIS A 1148 A method.

Series 2 was designed to investigate the effect of different compressive strength values on frost resistance of concrete at early ages and assess the deterioration behaviors in 300 freeze-thaw cycles. Before the freeze-thaw test, the pre-curing ages of specimens were 24 h, 44 h, 50 h, and 75 h, respectively. Measurement items were carried out after the pre-curing, then the specimens were subjected to 300 freeze-thaw cycles according to JIS A 1148 A method.

Series	Symbol	Pre-Curing Time Before Freezing (h)	Strength Development (MPa)	Recovery Curing Conditions	Experimental Items
-	Ν	-	-	-	
	F-T12	12	-		
Series 1	F-S5	24	5.0		
	F-S12	44	12.0	With recovery curing	Compressive strength,
	F-S18	50	18.0		Hydration degree,
	F-S25	75	25.0		Total porosity,
	F-S5	24	5.0		Freezing and thawing resistance
S	F-S12	44	12.0	Without recovery	
Series 2	F-S18	50	18.0	curing	
	F-S25	75	25.0		

Table 4.1 Experimental design of main experiments



Fig. 4.1 Flowchart of main experiments

In this study, several locations in cold regions from different countries were selected for the investigation to comprehensively evaluate the applicability of the minimum required compressive strength for cold weather concreting. The selected locations and meteorological data types are given in Table 4.2. The meteorological data used in the calculation were collected from the Integrated Surface Dataset (ISD) of the National Oceanic and Atmospheric Administration (NOAA). The five-year meteorological data from 2015 to 2019 were selected to obtain the average values.

Countries	Locations	Period (year)	Data types			
	Sapporo					
Japan	Obihiro					
	Morioka					
China	Beijing					
Cnina	Mohe					
Mongolia	Ulaanbaatar					
South Varias	Seoul		Annual extreme value			
South Korea	Busan		of daily minimum temperature, Number of days for			
	Vladivostok					
	Moscow	2015-2019				
Russia	Verkhoyansk		Total number of days			
	Anadyr		for freezing and			
	Nikel		thewing			
Sweden	Stockholm		unawing			
England	Edinburgh					
C 1	Calgary					
Canada	Iqaluit					
America	New York					
America	Fairbanks					

Table 4.2 Meteorological data collection program

### 4.2.2 Outdoor exposure tests

Table 4.3 shows the experimental design of the outdoor exposure tests. The symbol of the N specimen means the non-frozen concrete. The symbol of the F represents the frozen concrete. Experimental items in the laboratory tests were compressive strength, hydration degree, total porosity, mercury intrusion porosimetry (MIP), and freezing and thawing resistance, as shown in Table 4.3. The experimental test times for the outdoor exposure tests were initial, 0.5 years, 1 year and 3 years.

Symbol	w/c	Air content	Pre-curing before fre	g time ezing	Freezing and te thawing conditions		Experimental	Experimental items
	(70)	(%)	Temp.	Time	Temp.	Time	test times	Compressive strength,
			(°C)	(h)	(°C)	(h)		Hydration degree,
N					-		initial,	Total porosity,
F6	50	1.0	20	6	-20		0.5 year,	Mercury intrusion
F24		(Non-AE)	(Seal)	24	1↓ 5	72	1 year, 3 years	Freezing and thawing resistance

Table 4.3 Experimental design of main experiments

# 4.3 Experimental methods

# 4.3.1 Use Materials

# (1) Ordinary Portland Cement

Table 4.4 Chemical	composition	and physical	properties of OPC
--------------------	-------------	--------------	-------------------

		Ordinary Portland Cement		
		JIS Standard Value	Experimental Results	
Der	nsity (g/cm <sup>3</sup> )	-	3.17	
Specific S	urface Area (cm <sup>3</sup> /g)	More than 2500	3500	
	Water (%)	-	28.0	
Setting	Initial Setting (h $\cdot$ mm)	More than 60 min	1-45	
	Final Setting (h $\cdot$ mm)	Less than 10 h	3-00	
Soundness	Pat Test	Qualified	Qualified	
	1d	-	-	
Compressive	3d	12.5	32.9	
Strength	7d	22.5	51.0	
$(N/mm^2)$	28d	42.5	65.8	
	91d	-	-	
Heat evolution	7d	-	332	
(J/g)	28d	-	387	
Chemical	Magnesium Oxide	Less than 5.0	2.1	
Composition (%)	Sulfur Trioxide	Less than 3.5	1.9	

CHAPTER 4 EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT AT EARLY AGES ON FROST RESISTANCE OF CONCRETE

	Sulfur Dioxide	-	21.4	
	Aluminum Oxide	-	5.5	
	Ferric Oxide	-	2.8	
	Calcium Oxide	-	64.3	
	Ignition Loss	Less than 5.0	0.56	
	Total Alkali	Less than 0.75	0.25	
	Chloride Ion	Less than 0.035	0.012	
	Tricalcium Silicate	-	52	
Minoral	Dicalcium Silicate	-	23	
Mineral	Tricalcium Aluminate	-	10	
composition (70)	Tetracalcium		0	
	Aluminoferrite	-	9	

Ordinary Portland Cement was used in this experiment. Cement combines calcium, silicon, aluminum, iron, and other ingredients under close control. In this study, OPC was used in compliance with JIS standards. Table 4.4 shows the Chemical composition and physical properties of OPC.

#### (2) Aggregate materials

The physical properties of fine aggregates and coarse aggregates are shown in Table 4.5.

Types of aggregates	Surface dried density (g/cm <sup>3</sup> )	Absolute dried density (g/cm <sup>3</sup> )	Water absorption (%)	Coarse grain ratio (%)	Fineness modulus	Maximum size (mm)
Coarse	2.68	2.62	1.78	6.65	6.64	25
Fine	2.73	2.64	1.72	2.66	2.66	5.0

Table 4.5 Physical properties of aggregate materials

#### (3) Admixture

An air-entraining (AE) water-reducing agent (Master Pozzolith No. 70) was used to control air content in fresh concrete, and the target air content was  $4.5 \pm 1.5\%$ . An AE water-reducing agent is an admixture used to improve workability and resistance to freezing and thawing by entraining a large number of independent fine air bubbles

in concrete and reducing its unit water content. It has both the air entrainment action and the cement dispersing action of a water-reducing agent.

#### (4) Water

In this study, we used the tap water of our university.

# 4.3.2 Mix Proportions

#### (1) Laboratory tests

The concrete mix proportions of laboratory tests are listed in Table 4.6.

T 1 1 4 C	0	•		0	
Toblo / 6	( `onoroto	122137	proportiona	ot laborato	wr tooto
1 aDIC 4.0	CONCIER	IIIIX	DIODOLLIOHS	OF laborate	n v iesis
10010	001101000		propertions	01 100 01000	

	ala		Air Contont -	τ	U <b>nit Weig</b> l	AE Water-Reducing		
w/c	s/a (%)	Slump (cm)	(%)	W	С	S	G	Agent (mL/C = 100 kg)
0.5	47.1	$18\pm2.0$	$4.5\pm1.5$	175	350	852	957	250
		a	1 (0					

Note: W: water; C: cement; S: sand (fine aggregate); G: coarse aggregate.

#### (2) Outdoor exposure tests

Table 4.7 shows the concrete mix proportions of outdoor exposure tests.

				1 1		1			
			Unit weight	(kg/m <sup>3</sup> )			Absolute	volume	
w/c	s/a	W	OPC	S	G	W	OPC	S	G
50	42.4	210	420	747	994	210	133	279	378

Table 4.7 Concrete mix proportions of outdoor exposure tests

Note: W: water; C: cement; S: sand (fine aggregate); G: coarse aggregate.

# 4.3.3 Mixing Method

The fine aggregate and coarse aggregate were made surface dry on the day before the concrete was made, and the condition of the surface dry state was checked and adjusted on the day before use. The mixing was based on JIS R 5201-1997 "Physical test method of cement".

#### 4.3.4 Mold

The mold  $\varphi 100 \times 200$  (mm) cylinder-type plastic was used in the laboratory and outdoor exposure tests. The mold with dimensions of 75 × 75 × 400 mm was prepared for freeze–thaw test according to the JIS A 1148 A method.

#### 4.3.5 Test methods

#### 4.3.5.1 Fresh properties

The slump, air content, and temperature of fresh concrete were measured according to the JIS A 1101, JIS A 1128, and JIS A 1156, respectively.

#### 4.3.5.2 Hydration degree test

After the specified curing time, the samples were cut into 5 mm cube samples from the central area of the specimens. Acetone was used to stop the hydration process of samples for 1 day, and then the samples were dried using the F-drying method [41]. The sample was placed in a vacuum container for water absorption that exceeded 3 h and then put into a drying furnace at 105 °C for 24 h. After mass measurement, the sample was dried in a high temperature furnace at 1050 °C for 1.5 h. After cooling, the weight of the sample was measured. The hydration degree  $\alpha$  was calculated by the following equations:

Bound water content = 
$$\frac{m_1 - m_2}{m_2}$$

$$\alpha (\%) = \frac{Bound water content}{0.23}$$

where  $\alpha$  is the hydration degree (%);  $m_1$  is the sample mass after drying at 105 °C (g);  $m_2$  is the sample mass after drying at 1050 °C (g); 0.23 is the mass of water consumed when 1 g of cement is completely hydrated (g).

### 4.3.5.3 Underwater weighing test

The underwater weighing test was conducted to determine the total porosity of the concrete. Samples were

used as a whole specimen without cutting. After boiling the samples in hot water for 6 h, the fire was turned off, and the water was gradually cooled at room temperature. The samples were continued to be kept in the container for 24 h. Subsequently, samples were removed from the container, and the surface mois-ture was wiped so as to measure the mass of a saturated surface-dry specimen. The mass of the sample underwater was determined by putting the sample into the equip-ment for underwater weighing. And then, the sample mass of the absolute dry specimen was measured after drying at 105 °C for 24 h. The following equations were used to calculate the total porosity:

$$V_t(\%) = \left(1 - \frac{\rho_b}{\rho_{tr}}\right) \times 100\%$$
$$\rho_b \left(g/cm^3\right) = \frac{m_{ad}}{m_{sd} - m_w} \times \rho_w$$
$$\rho_{tr} \left(g/cm^3\right) = \frac{m_{ad}}{m_{ad} - m_w} \times \rho_w$$

where  $V_t$  is the total porosity (%);  $\rho_b$  is the bulk density (g/cm3);  $\rho_{tr}$  is the true density (g/cm3);  $m_{ad}$  is the sample mass of the absolute dry specimen (g) after drying at 105 °C;  $m_{sd}$  is the sample mass of saturated surface-dry specimen (g);  $m_w = m_t - m_{ew}$  is the mass of sample underwater (g);  $m_t$  is the mass of the equipment and sample underwater (g);  $m_{ew}$  is the mass of the equipment underwater (g);  $\rho_w$  is the density of water (g/cm<sup>3</sup>).

#### 4.3.5.4 Compressive strength test

The purpose of this test in order to determine of compressive strength of hardened concrete. This test was done as described in the Japanese Industry Standard A 1108. The compressive strength test was carried out using an Amsler's Compression Testing Apparatus. Fig. 4.2 shows the testing machine.

CHAPTER 4 EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT AT EARLY AGES ON FROST RESISTANCE OF CONCRETE



Fig. 4.2 Amsler's Compression Testing Apparatus

$$F_c = \frac{P}{A}$$

where,  $F_C$  is the compressive strength (N/mm<sup>2</sup>); P is the maximum applied force (N); A is the cross sectional area (mm<sup>2</sup>).

### 4.3.5.5 Freeze-thaw test

The freeze-thaw test was determined according to the JIS A 1148 A method on specimens with dimensions of  $75 \times 75 \times 400$  mm. One freeze-thaw cycle contains 4 h, which is a freezing condition of 2.5 h -18 °C and a thawing condition of 1.5 h +5 °C. The mass loss change and the fundamental transverse frequency of the specimens were measured within 300 cycles. The relative dynamic modulus of elasticity (RDM) and the durability factor (DF) were used to evaluate the resistance of concrete to freezing and thawing. The mass loss change, RDM, and DF of the specimens were calculated as follows:

$$W (\%) = \frac{w_0 - w_n}{w_0} \times 100\%$$
$$P_n(\%) = \left(\frac{f_n^2}{f_0^2}\right) \times 100\%$$
$$DF = \frac{P \times N}{M}$$

where W is the mass loss change (%);  $w_n$  is the specimen mass at n cycles (g);  $w_0$  is the specimen mass at 0

**CHAPTER 4** 

cycles (g);  $P_n$  is the relative dynamic modulus of elasticity (%);  $f_n$  is the fundamental transverse frequency at n cycles (Hz);  $f_0$  is the fundamental transverse frequency at 0 cycles (Hz); DF is the durability factor of concrete; P is the relative dynamic modulus of elasticity at n cycles; N is the number of cycles at which P reaches 60% or the number of cycles is 300, whichever is less; M is 300 cycles number.

#### 4.3.5.6 Mercury intrusion porosimetry (MIP) method

Many studies have been conducted to investigate the pore volume and pore size distribution of cement based materials with the mercury intrusion porosimetry (MIP) method, which was used as a typical way for the measurement of pore size distribution. MIP is based on the fact that a non-wetting fluid which has a contact angle greater than 90° for a particular solid will not intrude the pores of the solid without being pressurized. The pressure required depends on the pore shape, the surface tension and the contact angle of the liquid. The pore shape is generally assumed to be cylindrical for sedimentary rocks and sheet-like for crystalline rocks by Katsube and Williamson (1994). For pores of cylindrical shape, the relationship between the pore size and the applied pressure is given by Washburn (1921) according to the Young and Laplaces work on interfacial tension for the rise or depression of liquids in capillaries, as shown in the following equation:

$$r = \frac{-2\gamma\cos\theta}{P}$$

where, *r* is the pore radius (m), *P* is the pressure applied on mercury to intrude the pore  $(N/m^2)$ ,  $\gamma$  is the the surface tension between mercury and the pore wall (N/m),  $\theta$  is the the contact angle between mercury and the pore wall (degree). Based on the equation, the pore size distribution was derived from the applied pressure and the volume of mercury intruded into the pores. The surface tension and the contact angle for mercury adapted are equal to 0.484 N /m and 140°, respectively. The specific surface area of pores, dry density and porosity can be calculated by using the pore size distribution. The MIP techniques requires that the hardened cement – based materials should be thoroughly treated to remove water and evacuated prior to testing. After the 28 days curing age, mortar prisms were cut into 5 mm cube samples, and then dried to stop the hydration reaction by D-drying (ore Dry – ice drying) pretreatment. The samples pore size from 6 nm to 200000 nm was measured by a Autopore Master 33 porosimeter, Japan.

#### 4.3.5.7 *Cy<sub>ASTM-sp</sub>* Estimation Method

It is well known that  $Cy_{ASTM-sp}$  can estimate the freeze-thaw actions that concrete structures have received per year from the natural environment based on meteorological factors. The following equations were used to calculate the  $Cy_{ASTM-sp}$  results:

```
Cy_{ASTM-sp} = C \times F \times s \times p \times R_{a90}T = -ta \min (1 - Df/Dw)R_{a90} = 4.2T - 5.4
```

where  $Cy_{ASTM-sp}$  is the ASTM equivalent number of cycles with consideration of environmental coefficient (cycles per year); *C* is the curing conditions; *F* is the freeze-thaw conditions; *s* is the insolation conditions; *p* is the deterioration process coefficient;  $R_{a90}$  is the ASTM equivalent number of cycles based on the air temperature (cycles per year), it also means the deterioration process at a degree where the relative dynamic modulus of elasticity is at 100% to 90%; *T* is the regional coefficient; *ta* min is the annual extreme value of daily minimum temperature (°C); *Df* is the number of days for freezing, the number of days that the daily temperature continues to be below 0 °C; *Dw* is the total number of days for freezing and thawing, the number of days that the minimum temperature of the day is below -1 °C and the maximum temperature of the day exceeds 0 °C.

In addition, because the winter meteorological data were mainly used in this study, the calculation conditions were considered as insolation condition of the northern side (s), air curing condition (C), air freezing-water thawing condition (F), and deterioration process coefficient of 100% to 90% (p). Thus, according to the study content of Hama et al. (1999), these environmental coefficient values were determined. that is, s, C, F, and p were 1.00, 0.66, 0.21, and 1.00, respectively.

### 4.4 Experiment result and discussion

#### 4.4.1 laboratory tests

#### 4.4.1.1 Pre-experiment

Fig. 4.3 shows the results of determining the actual compressive strength and curing time. The results showed that all types of specimens achieved the expected compressive strength. Accordingly, the results of the curing time determined in the pre-experiment can be used to set the curing time for each type of specimen in the main experiments. The curing time of S5 ( $T_{S5}$ ),  $T_{S12}$ ,  $T_{S18}$ , and  $T_{S25}$  were 24 h, 44 h, 50 h, and 75 h, respectively.





#### 4.4.1.2 Main experiments

#### (1) Fresh concrete

Due to the limitations of the experimental conditions, the concrete specimens of Series 1 and 2 were manufactured in two batches. Table 4.8 presents the results of the slump, air content, and temperature of fresh concrete. It can be found that the fresh concrete in Series 1 and 2 obtained the target range of the slump and air content. The air content was kept at approximately 4.5% by using the AE water-reducing agent. In addition, fresh concrete of Series 1 and 2 exhibited good workability.

Table 4.8 Test results of fresh concrete

Series	Slump (cm)	Air content (%)	Temperature (°C)
Series 1	18.5	4.3	18.5
Series 2	17.5	4.0	19.0

#### (2) Compressive strength

It is necessary to determine whether the specimens attain the expected compressive strength values before performing the test items. The compressive strength test was measured for F-S5, F-S12, F-S18, and F-S25 in

Series 1 and 2. Fig.4.4 shows the determination result of the compressive strength for each type of specimen. It can be seen that the compressive strength values for each type of specimen in Series 1 and Series 2 met the experimental design requirements considering the dispersion.



Fig. 4.4 Determination of compressive strength after specified curing time

Fig. 4.5 shows compressive strength after recovery curing in Series 1. The compressive strength of all types of frozen (F) concrete specimens was roughly the same as that of non-frozen (N) concrete specimen. Especially, the compressive strength of F-T12 was only 1.1MPa before freezing, which was not met the requirement of the minimum required compressive strength. However, after recovery curing, the strength development was not slowed down. The compressive strength also reached the same degree as that of N. It can be considered that F-T12 was not suffered from the early age frost damage. The strength development of F-T12 was not affected by freezing because the AE water-reducing agent was added to the concrete, which ensures sufficient air content and effectively improves the early freezing resistance of concrete. This result is consistent with the findings of Yamashita (2021), who reported that the strength development of concrete with an AE agent at early ages is not affected by freezing after the final setting because the air content produced a significant effect.



Fig. 4.5 The compressive strength after recovery curing in Series 1

#### (3) Hydration degree and total porosity

Fig. 4.6 shows the results of the hydration degree and the total porosity in Series 1 at 31 days. Even if the compressive strength values of F specimens were approximately at the same extent as N after the recovery curing, the hydration degree results of F specimens were lower than that of N, and the total porosity results of F specimens were greater than that of N. It was found that if the pre-curing time of concrete is short, the hydration degree tends to be low, the total porosity tends to increase, and the effect of early age freezing remains. This result is consistent with the previous study (Bai et al., 2020), which revealed that early age frost damage could reduce the hydration degree of pure cement paste. In addition, although there is a difference in the hydration degree and the total porosity between F specimens and N specimen after the recovery curing, the difference is a little bit, which indicates that recovery curing enabled frozen concrete to achieve a comparable pore structure to that of non-frozen concrete.



Fig. 4.6 Results of hydration degree (a) and total porosity (b) in Series 1 at 31 days

The results of the hydration degree and the total porosity in Series 2 before the freeze-thaw test are shown in Fig. 4.7. From F-S5 to F-S25, the hydration degree was increased gradually, and the total porosity was decreased step by step. It showed an increased tendency in the hydration degree and a decreased tendency in the total porosity with the increase of curing time. However, although F-S25 had a compressive strength of more than 25.0 MPa, it still had much diffidence compared to N on the hydration degree and the total porosity. It illustrated that the hydration reaction of cement was not carried out completely at 75 h.



Fig. 4.7 Results of hydration degree (a) and total porosity (b) in Series 2 before freeze-thaw test

#### (4) Frost resistance of concrete

Fig. 4.8 shows the results of RDM and weight loss after 300 freeze-thaw cycles in Series 1. As can be seen in

**CHAPTER 4** 

Fig. 4.8, the results of RDM and mass change between F specimens and N were almost the same tendency until 300 cycles. Therefore, the durability factor results of all specimens in Series 1 were also at the same level, as shown in Fig. 4.9.

According to Fig. 4.5 and Fig. 4.6, in terms of compressive strength, hydration degree, and total porosity, the results of F specimens were near that of N, which demonstrated that the recovery curing could repair the internal deterioration caused by freeze-thaw cycles at early ages. In addition, according to the results of Table 4.8, the air content of fresh concrete was 4.3% in Series 1. Meanwhile, Hu et al. (2019) reported that concrete suffered from freezing after the final setting time that would lose little service performance of concrete. The dense matrix of hydration products was formed to resist the dilation pressure caused by the ice lens. Generally, when using Ordinary Portland cement, the final setting of fresh concrete is completed around 8 h, considering different w/b ratios (Hu et al., 2019; Yamashita, 2021). In Series 1, the pre-curing time periods of F specimens before exposure to early age freezing were 12 h, 24 h, 44 h, 50 h, and 75 h, respectively. The pre-curing time periods of all F specimens were obviously more than 8 h. From discussed above, the recovery curing, the freezing after the final setting of resistance to freezing after the final setting of N.



Fig. 4.8 Results of relative dynamic modulus of elasticity (a) and weight loss (b) after 300 freeze-thaw cycles in Series 1



Fig. 4.9 Durability factor results of Series 1

The results of the RDM and weight loss in Series 2 are shown in Fig. 4.9. It can be seen in Fig. 4.10, the RDM of F specimens decreased more rapidly compared to the reference specimen. The concrete with lower compressive strength showed early frost damage with the increasing number of freeze-thaw cycles. Besides, the weight loss tendency was similar to the RDM of concrete.

According to Fig. 4.7, different compressive strength values of concrete represented different denseness degrees of the pore structure inside concrete at early ages. When subjected to repeated freeze-thaw cycles, the aqueous solution in the capillaries and air voids produces dilation pressure due to freezing. Micro-cracks were caused when the strength of the skeleton structure was exceeded (Liu et al., 2011). As the number of freeze-thaw cycles increases, the micro-cracks inside concrete continually develop. Thus, in Fig. 4.10 (a), the greater the compressive strength of concrete, the higher the corresponding hydration degree, the denser the matrix of hydration products, the more freezing-thawing cycles it can withstand, and vice versa.



Fig. 4.10 Results of relative dynamic modulus of elasticity (a) and weight loss (b) after 300 freeze-thaw cycles in Series 2

Fig. 4.11 shows the relationship between the compressive strength and durability factor. It can be found that the durability factor increases with the increasing compressive strength. The value of  $R^2$  was greater than 0.9, which implied that there is a high positive correlation between compressive strength and durability factor. Besides, the compressive strength of N can also represent that of all F specimens in Series 1 because they had almost the same degree of compressive strength as that of N. Hence, it was demonstrated that the frost resistance of concrete at early ages depends on the pre-curing time and the hydration degree.



Fig. 4.11 Relationship between compressive strength and durability factor

From the above results, it was confirmed that air-entrained concrete could withstand several freeze-thaw

cycles at early ages and prevent early age frost damage by securing a compressive strength of 5.0 MPa. However, because the hydration reaction process of cement, which is during the compressive strength development at early ages, was not sufficiently advanced, the frost resistance of concrete at early ages was low. On the other hand, the freeze-thaw action from the natural environment in the cold weather concreting period or winter cannot cause damage equivalent to the 300 freeze-thaw cycles in JIS A 1148 A test method. Concrete structures after construction in winter would be expected to get a recovery curing from spring to autumn.

Besides, the numbers of freeze-thaw cycles that the RDM remained above 90% for F specimens in Series 2 were determined that F-S5 at 18 freeze-thaw cycles, F-S12 at 55 cycles, F-S18 at 90 cycles, and F-S25 at 124 cycles. According to the survey results of section 2 in this paper, the compressive strength of 5.0 MPa is used as the minimum required compressive strength for cold weather concreting in many countries. Therefore, 18 freeze-thaw cycles of F-S5 would be regarded as the reference values of deterioration occurrence to compare with the estimation results of  $Cy_{ASTM-sp}$  in this study.

#### (5) $Cy_{ASTM-sp}$ estimation based on winter meteorological factors

Table 4.9 shows the calculation results of winter meteorological data of various locations from different countries according to the Integrated Surface Dataset (ISD) of NOAA.

		Data types					
Countries Locations		Annual extreme value of daily minimum temperature (ta min, °C)	Number of days for freezing (Df, day)	Total number of days for freezing and thawing (Dw, day)			
	Sapporo	-11.92	43.20	106.2			
Japan	Obihiro	-43.08	48.00	137.4			
	Morioka	-10.56	7.00	100.8			
China	Beijing	-8.80	12.40	95.2			
Cnina	Mohe	-42.52	143.00	218			
Mongolia	Ulaanbaatar	-23.20	127.00	216.2			
South	Seoul	-14.22	19.80	80.6			

Table 4.9 Calculation results of winter meteorological data of various locations

CHAPTER 4 EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT AT EARLY AGES ON FROST RESISTANCE OF CONCRETE

Korea	Busan	-8.20	0.60	40.4
Russia	Vladivostok	-21.74	100.20	129.4
	Moscow	-15.40	66.00	106.6
	Verkhoyansk	-55.40	194.60	240.2
	Anadyr	-37.40	164.60	189.6
	Nikel	-30.20	133.60	161.8
Sweden	Stockholm	-12.30	25.20	63.4
England	Edinburgh	-6.06	0.60	24.4
Canada	Calgary	-28.20	55.60	135.6
	Iqaluit	-38.00	202.80	220.8
America	New York	-15.68	13.60	45.6
	Fairbanks	-34.24	133.20	192

 $Cy_{ASTM-sp}$  results of various locations were estimated as shown in Fig. 4.12.  $Cy_{ASTM-sp}$  results of all locations were lower than 18 freeze-thaw cycles. It was illustrated that the minimum required compressive strength of 5.0 MPa has sufficient frost resistance to protect concrete from early age frost damage during the cold weather concreting periods and winter. Therefore, according to the results of Fig. 4.12, it verified that the compressive strength of 5.0 MPa as the minimum required compressive strength for cold weather concreting is practicable. Before exposure to the freeze-thaw cycles, if the concrete is cured to more than 5.0 MPa, the quality of concrete may be guaranteed. As for some concrete constructions under critical water-saturated environments, such as dams and bridges, it is necessary to select a compressive strength value greater than 5.0 MPa to prevent damage due to freeze-thaw cycles, depending on the construction requirements.

In this study, the applicability of 5.0 MPa as the minimum required compressive strength for cold weather concreting was determined for the first time with the combination of experiments and estimation based on meteorological factors. Moreover, the findings of this study were persuasive by investigating guidelines of cold weather concreting in different countries and winter meteorological data of various locations in the world.



Fig. 4.12 Cy<sub>ASTM-sp</sub> results of various locations

# 4.4.2 Outdoor exposure tests

# 4.4.2.1 Compressive strength

Fig. 4.13 shows the compressive strength for outdoor exposure tests of change over time. It can be seen that the compressive strength of all samples increases with time, and the compressive strength reaches the limit degree at 1 year of exposure time. Strength development does not continue to increase at 3 years of exposure time. Among them, the compressive strength values of N and F24 were almost the same degree, and F6 was obviously affected by the early age frost damage, and the compressive strength ratio with N was lower than 90%.



Fig. 4.13 Compressive strength for outdoor exposure tests of change over time

Fig. 4.14 shows the compressive strength of standard curing at 28 days. The compressive strength of specimens of N and F24 was the same degree. It also means that F24 was not affected by early age frost damage. The compressive strength of F6 specimens was significantly lower than that of N, and the compressive strength ratio with N was lower than 90%. It can be seen that F6 was subjected to early age frost damage.



Fig. 4.14 Compressive strength of standard curing at 28 days

# 4.4.2.2 Hydration degree

Fig. 4.15 shows the hydration degree of change over time. The hydration degree of N, F6, and F24 had the same trend with increasing time. At 1 year of exposure time, the hydration degree tends to be the maximum value. The hydration degree at 3 years of exposure time remained at the same degree as 1 year of exposure time. The trend of hydration degree is the same as the trend of compressive strength.



Fig. 4.15 Hydration degree of change over time

# 4.4.2.3 Total porosity

Fig. 4.16 shows the total porosity of change over time. It can be seen that the hydration degree increases, and the total porosity decreases with time. The total porosity decreases to the minimum value at 1 year of exposure time.



Fig. 4.16 Total porosity of change over time

# 4.4.2.4 Mercury intrusion porosimetry (MIP)

Fig. 4.17, 4.18, and 4.19 show the result of (a) cumulative pore volume and (b) pore distribution of N, F6, and
F24 within 3 years. By observing each (a) and (b), the pore structure shows a densification trend with the increase of time. It can be seen that N, F6, and F24 have the same variation in pore volume and pore size distribution. The sample of 1 year of exposure time has the densest pore structure. Also, the sample of 3 years of exposure time was slightly higher than 1 year of exposure time in (a) and (b). It is possible that the pore structure may have changed coarse because it experienced the influence of dry and wet cycles in 3 years.







Fig. 4.18 MIP analysis of (a) cumulative pore volume and (b) pore distribution of F6 within 3 years

#### CHAPTER 4 EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT AT EARLY AGES ON FROST RESISTANCE OF CONCRETE



Fig. 4.19 MIP analysis of (a) cumulative pore volume and (b) pore distribution of F6 within 3 years

Fig. 4.20, 4.21, and 4.22 show the result of (a) cumulative pore volume and (b) pore distribution of N, F6, and F24 from initial to 3 years of exposure time. At the initial exposure time, the sample of F6 has the coarsest pore structure. The samples of N and F24 have almost the same degree of pore structure. For the results of 1 year of exposure time, it can be seen that the pore structure of F6 was the loosest. It is considered that early age frost damage could cause the pore structure to become coarse. The samples of N and F24 also have the same degree of pore structure. At 3 years of exposure time, it can be found that the tendency of pore volume and pore size distribution was the same as that of 1 year of exposure time. The degree of denseness order in pore structure is  $F24 \le N < F6$ .



Fig. 4.20 MIP analysis of (a) cumulative pore volume and (b) pore distribution at initial exposure time

#### CHAPTER 4 EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT AT EARLY AGES ON FROST RESISTANCE OF CONCRETE



Fig. 4.21 MIP analysis of (a) cumulative pore volume and (b) pore distribution at 1 year of exposure time



Fig. 4.22 MIP analysis of (a) cumulative pore volume and (b) pore distribution at 3 years of exposure time

#### 4.4.2.5 Frost resistance of concrete

Fig. 4.23 shows the results of (a) RDM and (b) weight loss at initial exposure time. It can be found that the decreasing trend of RDM for F24 and F6 is obvious, with a significant difference compared to N. Because at the initial exposure time, the compressive strength of F24 and F6 was low, and the internal pore structure was not dense enough. Therefore, the ability to resist freezing and thawing was poor.

CHAPTER 4 EFFECT OF COMPRESSIVE STRENGTH DEVELOPMENT AT EARLY AGES ON FROST RESISTANCE OF CONCRETE



Fig. 4.23 Results of relative dynamic modulus of elasticity (a) and weight loss (b) at initial exposure time

Fig. 4.24 shows the results of (a) RDM and (b) weight loss after recovery curing time. It can be found that the RDM of F24 reached the same level as that of N after recovery curing. The frost resistance of F6 remained low even after recovery curing because it suffered from early age frost damage. Since it is Non-AE concrete, the frost resistance is worse than that of AE concrete, and the RDM of both N and F24 droped below 60% within 300 freeze-thaw cycles.



Fig. 4.24 Results of relative dynamic modulus of elasticity (a) and weight loss (b) after recovery curing time

Fig. 4.25 shows the results of (a) RDM and (b) weight loss at 3 years of exposure time. It can be found that the RDM of F24 reached the same level as that of N after 3 years exposure time. The frost resistance of F6 still remained low degree because it suffered from early age frost damage. Since it is Non-AE concrete, the frost resistance is worse than that of AE concrete, and the RDM of both N and F24 drops below 60% within 300 freeze-thaw cycles.



Fig. 4.25 Results of relative dynamic modulus of elasticity (a) and weight loss (b) at 3 years of exposure time

Fig. 4.26 shows the RDM results of N at different exposure times. From the RDM values of 28 days and 3 years of exposure time, Non-AE concrete has low frost resistance due to a lack of sufficient air content in concrete. The RDM values were still below 60% within 300 freeze-thaw cycles, even after the standard or longer curing period. In addition, the RDM results of 3 years of exposure time were lower than that of 28 days. The reasons can be considered in the following two aspects: first, it can be known that the freezing and thawing cycles in one year are two or three cycles in most cold regions according to Fig. 4.11, so 3 years of exposure time could obtain about ten cycles. The frost resistance could be deceased because the samples have already experienced freezing and thawing cycles under outdoor exposure tests. Second, the dry and wet cycles from summer to winter also can cause the frost resistance of concrete to be reduced.



Fig. 4.26 Results of relative dynamic modulus of elasticity of N at different exposure times

Fig. 4.27 shows the RDM results of F6 at different exposure times. It can be found that the frost resistance of F6, which has been subjected to the early age frost damage, shows a significantly decreased trend compared with that of N after recovery curing or a longer period. Also, the RDM results of 3 years of exposure time were slightly lower than 31 days. It is considered that the long-term exposure environment affects the frost resistance of concrete.



Fig. 4.27 Results of relative dynamic modulus of elasticity of F6 at different exposure times

Fig. 4.28 shows the RDM results of F24 at different exposure times. It can be seen that the RDM results of F24 at 31 days were the same degree as that of N at 28 days. It also means that F24 samples were not subjected to the early age frost damage. Similarly, the RDM results of 3 years of exposure time were lower than 31 days. According to Fig. 4.25, Fig. 4.26, and Fig. 4.27, it can be found that the RDM results of outdoor exposure tests were below that of laboratory curing tests.



Fig. 4.28 Results of relative dynamic modulus of elasticity of F24 at different exposure times

#### 4.5 Conclusion

In this chapter, the experimental program was designed into two parts to assess the effect of compressive strength development at early ages on the frost resistance of concrete in both laboratory tests and outdoor exposure tests. Meanwhile, the applicability of minimum compressive strength for cold weather concreting was evaluated according to meteorological factors. The main findings of this chapter were shown as follows:

(1) Air-entrained concrete that reaches a compressive strength of 5.0 MPa can withstand several freeze-thaw cycles and effectively prevent early age frost damage.

(2) For early-age concrete subjected to repeated freeze-thaw cycles in a critical water-saturated environment, the frost resistance increases with the increase of compressive strength. For OPC concrete with w/c of 0.5, the numbers of freeze-thaw cycles of the concrete with compressive strength of 5.0, 12.0, 18.0, and 25.0 MPa that

can maintain the relative dynamic modulus of elasticity above 90% were about 18, 55, 90, and 124 cycles, respectively.

(3) Concrete that has finished its final setting can effectively resist early age frost damage not only in Air-

entrained condition but also in Non-air-entrained condition.

(4) The frost resistance of concrete in outdoor exposure tests was lower than that of concrete in laboratory

tests. The dry-wet and freeze-thaw cycles in the natural environment can reduce the frost resistance of concrete.

## **References:**

Powers, T.C. Prevention of frost damage to green concrete. RILEM Bull. 1962, 14, 120-124.

George, C.H.; Alan, D.B. Considerations in the prevention of damage to concrete frozen at early ages. J. Am. Concr. Inst. 1983, 80, 371–376.

Moller, G. Tests of resistance of concrete to early frost action. In RILEM Symposium on Winter Concreting, Theory and Practice; Session B-1; p. 31; RILEM: Copenhagen, Denmark, Feb. 1956.

Yi, S.; Pae, S.; Kim, J. Minimum curing time prediction of early-age concrete to prevent frost damage. Constr. Build. Mater. 2011, 25, 1439–1449.

Koh, Y. A Study on Winter Construction of Concrete in Hokkaido. Ph.D. Thesis, Hokkaido University, Hokkaido, Japan, 1959. (In Japanese)

Recommended Practice for Cold Weather Concreting; American Concrete Institute Committee 306-66; American Concrete Insti-tute: Farmington Hills, MI, USA, 1966.

Recommendations for Winter Concreting; RILEM Bulletin No. 21; Winter Construction Committee, RILEM: Copenhagen, Den-mark, 1963.

Voellmy, A. High Concrete Quality in Cold Weather. In RILEM Symposium on Winter Concreting; Session D; RILEM: Copen-hagen, Denmark, Feb. 1956.

Rastrup, E. Requirdening Time by Freezing of Green Concrete in Relation to the Cements; RILEM Bulletin No. 23; Recommenda-tions for Winter Concreting; RILEM: Copenhagen, Denmark, 1964.

Koh, K.T.; Park, C.J.; Ryu, G.S.; Park, J.J.; Kim, D.G.; Lee, J.H. An experimental investigation on minimum compressive strength of early age concrete to prevent frost damage for nuclear power plant structures in cold climates. Nucl. Eng. Technol. 2013, 45, 393–400.

Choi, H.; Zhang, W.; Hama, Y. Method for determining early-age frost damage of concrete by using airpermeability index and influence of early-age frost damage on concrete durability. Constr. Build. Mater. 2017, 153, 630–639.

Guide to Cold Weather Concreting; American Concrete Institute Committee 306; American Concrete Institute: Farmington Hills, MI, USA, 2016.

C666/C666M-15; Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing. ASTM International: West Conshohocken, PA, USA, 2015.

JIS A 1148; Method of Test for Resistance of Concrete to Freezing and Thawing. Japan Standards Association: Tokyo, Japan, 2010. (In Japanese).

GB/T50082-2009; The Test Method of Long-Term and Durability on Ordinary Concrete. Industry Standard of the People's Republic of China: Beijing, China, 2009. (In Chinese)

Hasegawa, K.; Hon, E. Influence of External Factors on Frost Damage of Concrete and Frost Damage Risk in Japan; Research Re-port; Faculty of Engineering, Hokkaido University: Hokkaido, Japan, 1979. (In Japanese)

Hama, Y.; Matsumura, K.; Tabata, M.; Tomiita, T.; Kamada, E. Estimation of frost damage of concrete based on meteorolog-ical factors. J. Struct. Constr. Eng. 1999, 523, 9–16.

Quy, N.X.; Noguchi, T.; Na, S.; Kim, J.; Hama, Y. Distribution map of frost resistance for cement-based materials based on pore structure change. Materials. 2020, 13, 2509. https://doi.org/10.3390/ma13112509.

Standard Specifications for Concrete Structures, Materials & Construction; Japan Society of Civil Engineers: Tokyo, Japan, 2017. (In Japanese)

RILEM recommendations for concreting in cold weather; VTT Technical Research Centre of Finland: Espoo, Finland, 1988.

Concrete Materials and Methods of Concrete Construction/Test Methods and Standard Practices for Concrete; Canadian Stand-ards Association: Toronto, ON, Canada, 2014.

Specification for Winter Construction of Building Engineering; Ministry of Housing and Urban-Rural Development of the Peo-ple's Republic of China: Beijing, China, 2011. (In Chinese)

Recommendations for the Production of Concrete Work in Winter; Non-profit partnership self-regulatory organization union of construction companies of Ural and Siberia: Chelyabinsk, Russia, 2015. (In Russian)

Concrete Standard Specification Ministry of Land; Infrastructure and Transport of South Korea: Seoul, South Korea, 2016. (In Korean)

Saul, A.G.A. Principles Underlying the Steam Curing of Concrete at Atmospheric Pressure; Magazine of Concrete Research: London, UK, 1951; Volume 2, pp. 127–140. https://doi.org/10.1680/macr.1951.2.6.127.

Koh, Y. Proposal of practical method for estimating concrete strength and its application (material/construction). Archit. Inst. Jpn. 1959, 63, 129–132.

Integrated Surface Dataset (Global). National Centers for Environmental Information of National Oceanic and Atmospheric Administration. Available online: https://www.ncei.noaa.gov/access/search/data-search/global-hourly (accessed on 5 Octo-ber 2020).

Yamashita, K. Mechanism of Strength Enhancement Stagnation of Concrete Subjected to Freezing during the Setting and Hardening Process and Measurement of the Extent and Depth of Frost Damage. Ph.D. Thesis, Muroran Institute of Technolo-gy, Hokkaido, Japan, 2021. (In Japanese). http://doi.org/10.15118/00010421.

Bai, S.; Guan, X.; Li, G. Effect of the early-age frost damage and nano-SiO2 modification on the properties of Portland cement paste. Constr. Build. Mater. 2020, 262, 120098.

Hu, X.; Peng, G.; Niu, D.; Zhao, N. Damage study on service performance of early-age frozen concrete, Constr. Build. Mater. 2019, 210, 22–31.

Liu, L.; Ye, G.; Schlangen, E.; Chen, H.; Qian, Z.; Sun, W.; Breugel, K.V. Modeling of the internal damage of saturated cement paste due to ice crystallization pressure during freezing. Cem. Concr. Compos. 2011, 33, 562–571.

# CHAPTER 5

# INVESTIGATION OF THE EFFECT OF PREVENTING EARLY AGE FROST

# DAMAGE USING ADDITIVE FOR SETTING TIME ADJUSTMENT

#### 5.1 Overview

In Chapter 4, the compressive strength result of F-T12, which was measured in the laboratory tests, shows that even though the compressive strength was lower than 5 MPa, the F-T12 samples were not affected by the early age frost damage after the recovery curing time. In addition, the frost resistance of F-T12 has the same degree as that of N. It is kept in correspondence with the investigation of Yamashita (2021). He reported that the strength development of concrete with an AE agent at early ages is not affected by freezing after the final setting because the air content produced a significant effect. Meanwhile, the compressive strength result of F24, a Non-AE concrete sample tested in the outdoor exposure tests, presents that it was not subjected to the early age frost damage after the recovery curing time. The pre-curing times before early age freezing of F-T12 and F24 are 12 hours and 24 hours, respectively. It means that both F-T12 and F24 have finished the final setting before suffering from early age freezing.

Therefore, it is considered that if the placed concrete could finish the final setting at the initial hardening stage, the concrete is possible to resist early age freezing. If the setting time of concrete could be controlled in cold weather concreting, it would protect the placed concrete from early age frost damage.

However, it is well-known that the setting time of concrete is delayed in low-temperature environments. Recently, an additive for setting time adjustment has been developed to reduce the setting time and improve the efficiency of the work by shortening the time required for surface finishing and improving the concrete quality. This concrete additive is recommended to apply in the finishing work during cold season, and using this additive for setting time adjustment method is named Advanced Concrete Finish (ACF) Construction method. Relevant studies (Ishii et al. 2018; Ishii et al. 2019) on this additive have investigated the effects of shortening the setting time and reducing bleeding by mixing a predetermined amount of additive into concrete and comparing them with the case of concrete without additive.

As a result, it was confirmed that the fresh properties were secured, bleeding was reduced, and the setting time was accelerated under  $10^{\circ}$ C. In addition, the tests in the construction site showed that the addition rate of additive for setting time adjustment in the range of 2.5 to 4 kg/m<sup>3</sup> contributed to a reduction in working time of

approximately 3 to 5 hours. However, there is still a lack of investigation into the effectiveness of preventing early age frost damage using the ACF additive.

Consequently, there is a new technology development to prevent early age frost damage by using the ACF additive for setting time adjustment in Chapter 5. The investigation of ACF additive mainly explored the fresh properties of concrete, the setting time, compressive strength development, and prevention effectiveness of early age frost damage under different addition amount conditions. The objective of Chapter 5 is to (1) investigate the effect of shortening the setting time of concrete at room temperature and low temperature using different amounts of ACF additive, (2) examine the effect on strength development under different amounts, and (3) propose a suitable use program of ACF additive based on the prevention effectiveness of early age frost damage.

## 5.2 Experimental design

#### 5.2.1 Experimental program

To investigate the effect of different amounts of ACF additive on the setting time and compressive strength, four concrete types of different amounts of ACF additive were mixed to make concrete test cylindrical plastic molds with dimensions of  $\emptyset 100 \times 200$  mm. The additive amount of ACF additive had four levels: 0, 2, 4, and 6 kg/m<sup>3</sup>, as shown in Table 5.1. The concrete samples were put into a room with 20°C and 60% relative humidity and a room with 5°C and 60% relative humidity for curing until 28 days and 31 days. All samples were conducted in sealed condition. The experimental items include slump, air content, setting time, and compressive strength.

T 11 7 1	<b>F</b> • • • • •	1 .	C	•	
Toble S L	Hynorimontal	dacion	ot avi	norim	anto
Table J.L	EXDUITIONIAL	ucsign			LIILO
	r				

Symbol	Cement	Additive	Addition amount (kg/m <sup>3</sup> )	w/c	Slump (cm)	Air content (%)	Experimental items
Р			0	0.5	18±2.0	4.5 <u>+</u> 1.5	Slump,
A2	ODC	ACF	2				Air content,
A4	OPC		4				Setting time,
A6	A6		6				Compressive strength

Fig. 5.1 shows the flowchart of experiments. Concrete samples were placed in both a 20°C temperature room and a 5°C temperature room and cured in a sealed condition at early ages. A setting time test was conducted on non-frozen (N) concrete samples from each concrete designed in this experiment after placement. The compressive strength tests were conducted at 1, 3, 7, 28, and 31 days. For frozen concrete, the samples were precured for 6, 12, and 24 hours. Frozen concrete samples were transferred to an adjustable temperature chamber and exposed to 3 freeze-thaw cycles in air, with each cycle consisting of 12 hours at -20 °C followed by 12 hours at +5 °C. Fresh concrete is highly susceptible to frost damage under these freeze-thaw conditions due to excess free water in the concrete. After undergoing freeze-thaw cycles in air for 3 cycles, the samples were returned in a room with a temperature of 20°C and a relative humidity of 60% to carry out the recovery curing until 31 days.



Fig. 5.1 Flowchart of experiments

## 5.3 Experimental methods

#### 5.3.1 Use Materials

#### (1) Ordinary Portland Cement

Ordinary Portland cement based on JIS R 5210 in Japan was used in this experiment (Nippon steel cement corporation, Muroran, Japan), and its specific gravity was 3.17 g/cm<sup>3</sup>.

#### (2) Aggregate materials

Table 5.2 displays the physical properties of both fine and coarse aggregates.

Types of aggregates	Surface dried density (g/cm <sup>3</sup> )	Absolute dried density (g/cm <sup>3</sup> )	Water absorption (%)	Coarse grain ratio (%)	Fineness modulus	Maximum size (mm)
Coarse	2.66	2.62	1.71	6.65	6.64	25
Fine	2.64	2.63	1.75	2.66	2.66	5.0

#### (3) Admixture

Controlling the air content in fresh concrete was achieved using the AE agents of Master Pozzolith 78S and Master Air 202, and the target air content was  $4.5 \pm 1.5\%$ .

DENKA ACF-W is a field-added setting accelerator that controls the setting time of concrete. By eliminating setting time delays caused by low temperatures and reducing waiting time for floor finishing, DENKA ACF-W not only improves productivity but also reduces quality degradation caused by continuous bleeding.

#### (4) Water

In this study, we used the tap water of our university.

## 5.3.2 Mix Proportions

Table 5.3 Concrete mix proportions										
	-   -			Unit Weight (kg/m <sup>3</sup> )			AE Agent		ACE W	
w/c	s/a	Siump	Air Content-	W	C	S	C	78S	202	AUF-W
	(70)	o) (cm)	(70)	w c	C	C S	U	(C×wt.%)	(C×wt.%)	(kg/m)
0.5	47.1	$18\pm2.0$	$4.5\pm1.5$	175	350	832	941	0.55	0.004	0, 2, 4, 6

The concrete mix proportions are listed in Table 5.3.

Note: W: water; C: cement; S: sand (fine aggregate); G: coarse aggregate.

## 5.3.3 Mixing Method

Fine and coarse aggregate surface drying was performed the day before the concrete was poured. The surface dry state was inspected and adjusted on the day of use. The mixing was based on JIS R 5201-1997 "Physical test method of cement".

## 5.3.4 Mold

The mold  $\varphi$ 100×200(mm) cylinder-type plastic was used in the tests.

# 5.3.5 Test methods

## 5.3.5.1 Fresh properties

Fresh concrete was measured using the JIS A 1101, JIS A 1128, and JIS A 1156 standards, respectively, for the slump, air content, and temperature.

# 5.3.5.2 Compressive strength test

Compressive strength test was conducted using a Ø 100 × 200 mm cylinder specimen in accordance with JIS

A 1108.

## 5.3.5.3 Setting time test

The setting time test was measured according to JIS A 1147, "Method of test for time of setting of concrete

mixtures by penetration resistance". Concrete was poured into the resin mold formwork (Fig. 5.2) in a single layer up to about 1 cm from the top surface of the formwork, poked 15 times with a poker, lightly tapped the sides of the formwork to eliminate the poke holes, and the top surface was leveled with a trowel. The formwork was covered with a lid to prevent water from evaporating, and the lid was not removed except for removing bleeding water or for measurement. The measurement procedure is described below:

- ① Attach a penetration needle (Fig. 5.3) with a cross-section of 100 mm<sup>2</sup> to the measuring device (Fig. 5.4).
- <sup>(2)</sup> Lower the lever to the mark on the penetration needle and make a penetration in about 10 seconds. Record the time of the penetration test and the force (N) required for the penetration by reading from the device. If bleeding water is floating on the surface, remove the bleeding water with a dropper or the like before measurement. In the second and subsequent measurements, the penetration shall be made, avoiding the area disturbed in the previous penetration test, and the distance between the needle marks of the penetration needle shall be at least twice the diameter of the penetration needle used or at least 15 mm.
- ③ Repeat step ② until the penetration resistance exceeds 3.5 N/mm<sup>2</sup>. The procedure shall be repeated at least
  6 times until a penetration resistance value of 3.5 N/mm<sup>2</sup> is obtained.
- ④ When the penetration resistance exceeds 3.5 N/mm<sup>2</sup>, replace the penetration needle with one with a cross-section of 25 mm<sup>2</sup> (Fig. 5.3) and repeat the procedure in ② until the penetration resistance reaches 28 N/mm<sup>2</sup>.

The initial setting time of concrete is the time it takes for the resistance to penetration to reach 3.5 N/mm<sup>2</sup>, and the final setting time is the time it takes for the resistance to penetration to reach 28 N/mm<sup>2</sup>. The following equation can be used to determine the penetration resistance:

$$R(N/mm^2) = \frac{P(N)}{A(mm^2)}$$

where *R* is the penetration resistance value (N/mm<sup>2</sup>); *P* is the force required for penetration (N); *A* is the cross-section area of the penetrating needle used (mm<sup>2</sup>).



Fig. 5.2 Resin mold formwork



Fig. 5.3 Penetration needles (Left: cross-section 25 mm<sup>2</sup>, Right: cross-section 100 mm<sup>2</sup>)



Fig. 5.4 Measuring device

## 5.4 Results and discussion

## 5.4.1 Fresh concrete

Fig. 5.2 shows the slump test results of the fresh concrete samples. It is apparent from the figure that the slump values for all types of concrete samples fell within the target range of  $18 \pm 2.0$  cm. This finding suggests that the ACF additive did not cause a significant impact on the slump of the concrete immediately after placement.



Fig. 5.2 Slump of change over time

The data illustrated in Fig. 3 reveal that the air content of all concrete sample types met the target range of 4.5

 $\pm$  1.5%. It indicated that the use of the ACF additive did not exert any discernible influence on the air content of

the fresh concrete. Accordingly, the air content of the fresh concrete can remain stable and consistent even using the ACF additive.



Fig. 5.3 Air content of all concrete sample types

#### 5.4.2 Setting time of concrete

Fig. 5.4 shows the results of setting time for all concrete sample types under (a) 20°C temperature and (b) 5°C temperature conditions. For both 20°C temperature and 5°C temperature conditions, there is a common tendency that the more the amount of ACF additive, the faster the setting time of concrete.

Fig. 5.5 shows the setting time durations of all concrete sample types. The setting time duration differences between all types of ACF concrete and P sample in 20°C temperature condition were smaller than that of 5°C temperature condition. It indicates that the ACF additive is more effective in reducing the setting time of concrete under low temperature conditions, especially in the case of the A6 concrete sample, which has a much shorter setting time than other concrete samples.



Fig. 5.5 Setting time durations of all concrete sample types

Fig. 5.6 shows the relationship between addition amounts of ACF additive and final setting times. It can be found that with the increase addition amounts of ACF additive, the final setting time of concrete showed a decreasing trend in 20°C temperature and 5°C temperature conditions.

Meanwhile, according to the results of Fig. 5.4, Fig. 5.4, and Fig. 5.6, it is confirmed that the ACF additive has a practical effect on shortening the setting time of concrete and can also significantly shorten the setting time of concrete in a low temperature environment.



Fig. 5.6 Relationship between addition amounts of ACF additive and final setting times

#### 5.4.3 Compressive strength of concrete at early ages

The compressive strength results of all concrete samples at early ages in both 20°C and 5°C temperature conditions are shown in Fig. 5.7. It has been observed that there is little strength development in all concrete samples within the first 6 hours. As shown in Fig. 5.5, the samples were still in the process of setting, making it difficult to determine the strength development of the concrete at 6 hours. The compressive strength increases as the amount of ACF additive increases within 24 hours, both in 20°C and 5°C temperature conditions. It is evident that using ACF additive can enhance the compressive strength of concrete compared to that of concrete without ACF additive within 24 hours. Therefore, there is a difference in strength development during early ages up to 24 hours. As for the compressive strength results of 7 days, at 20°C temperature, the compressive strength results of ACF concrete samples were slightly higher than those of the P sample. In contrast, at 5°C temperature, there were no noticeable differences in compressive strength between the ACF concrete samples and the P sample at 20°C temperature, allowing them to progress into the hardening stage rapidly. This faster hardening is thought to account for the higher compressive strength values observed in the ACF concrete samples at early ages. In the case of the 5°C temperature condition, it is suggested that the low-temperature environment predominantly affects the strength development. As a result, the compressive strength results of the ACF concrete



samples and the P sample were not significantly different at 7 days under 5°C temperature.

Fig. 5.7 Compressive strength of all types of concrete samples at early ages

## 5.4.4 Compressive strength of concrete at 28 days

Fig. 5.8 shows the result of the compressive strength of all types of concrete samples at 28 days in 20°C temperature and 5°C temperature conditions. It can be found that all types of concrete samples have the same degree of compressive strength in both 20°C temperature and 5°C temperature conditions. The compressive strength values of all types of concrete samples at 20°C temperature were slightly larger than that at 5°C temperature. It can be seen that the curing temperatures affected the compressive strength of the concrete. The low temperature environment leads to the hydration degree of concrete is lower than that of a normal temperature environment. The result shows that the addition amounts of ACF additive do not influence the compressive strength at 28 days. Because the ACF additive is mainly to shorten the setting time of concrete, it can improve the compressive strength at 20°C temperature at early ages. However, it can not increase compressive strength for a long curing time. For ACF concrete, the compressive strength at 28 days depends on the mix proportion of concrete, materials, and curing temperature.



Fig. 5.8 Compressive strength of all types of concrete samples at 28 days

Fig. 5.9 shows the relationship between compressive strength development and maturity. Fig. 5.9 included all the compressive strength values of concrete samples in both 20°C temperature and 5°C temperature conditions. It is well known that a logistic curve can show all of the strength development processes, and it is widely used to estimate concrete strength due to its accuracy. It can be seen that all compressive strength results coincided exactly with the trend of the logistic curve. It also demonstrates that the ACF additive at any addition amount has no effect on the compressive strength development of concrete, and the strength development of concrete is not related to ACF additive.



Fig. 5.9 Relationship between the compressive strength development and maturity

#### 5.4.5 Prevention effectiveness of early age frost damage using ACF additive

Fig. 5.10 shows the results of the compressive strength of P frozen concrete samples after recovery curing. It can be found that the F6 samples were lower than the other samples. The compressive strength values of F12 and F24 samples have the same degree as that of P-20-N after recovery curing. It means that the F12 and F24 samples were not subjected to early age frost damage. In addition, it can be seen that the environment temperatures of pre-curing before the early age freezing have no effect on the compressive strength at 840 °D · D not only in F12 and F24 samples but also in F6 samples. The compressive strength of 20°C temperature pre-curing is approximately equal to that of 5°C temperature pre-curing.



Fig. 5.10 Compressive strength of P frozen concrete samples after recovery curing

Fig. 5.11 shows the result of compressive strength ratios based on P-20-N of all frozen concrete samples at 840 °D ·D. It can be seen that F6 samples were subjected to early age frost damage because the compressive strength ratios were below 90%. According to Fig. 5.5, the final setting time of P samples in 20°C temperature is elapsed time of 7:42, and the final setting time of P samples in 5°C temperature is elapsed time of 11:33, so F6 samples suffered from early age frost damage. The early age freezing time of the F12 and F24 samples was behind the final setting time. Even though the compressive strength values of F12 and F24 samples were lower than the minimum required compressive strength of 5.0 MPa when early age freezing occurred, the compressive strength ratios of F12 and F24 samples were almost 100% after recovery curing, which indicates that early age freezing did not affect F12 and F24 samples. It also demonstrates once again that the strength development of

concrete at early ages is not affected by freezing after the final setting, which is reported by Yamashita (2021)

and the findings of Chapter 4 in this thesis.



Fig. 5.11 Compressive strength ratios based on P-20-N of frozen concrete samples at 840 °D ·D

Fig. 5.12 presents the results of the compressive strength test on A2 frozen concrete samples after recovery curing. It can be observed that the compressive strength of F6 samples was lower than that of other samples. On the other hand, the compressive strength values of F12 and F24 samples were similar to that of A2-20-N after recovery curing, indicating that F12 and F24 samples did not suffer from early age frost damage. Moreover, the pre-curing temperature did not affect the compressive strength of F12, F24, and F6 samples at 840 °D ·D for freezing and thawing cycles. The compressive strength of concrete cured at 20°C was nearly the same as that of concrete cured at 5°C.

Fig. 5.13 illustrates the compressive strength ratios of all frozen concrete samples based on A2-20-N at 840 °C for freezing and thawing cycles. It is evident that the compressive strength ratios of F6 samples were less than 90%, indicating early age frost damage. Based on the findings in Fig. 5.5, the final setting time of A2 samples at 20°C is 6:49, and that of A2 samples at 5°C is 11:14, implying that F6 samples experienced early age frost damage. However, F12 and F24 samples were frozen after the final setting time. Although the compressive strength values of F12 and F24 samples were lower than the required minimum compressive strength of 5.0 MPa at early age freezing, their compressive strength ratios were almost 100% after recovery curing, indicating that

early age freezing did not impact F12 and F24 samples. This finding supports the previous studies conducted by Yamashita (2021) and Chapter 4 of this thesis, which showed that concrete strength development at early ages is not affected by freezing after the final setting.

However, based on the comparison of Fig. 5.11 and Fig. 5.13, it is clear that the preventive measures against early age frost damage in A2 concrete have not been achieved. A2 concrete has the same vulnerability to early age frost damage as P concrete.



Type of specimen

Fig. 5.12 Compressive strength of A2 frozen concrete samples after recovery curing



Fig. 5.13 Compressive strength ratios based on A2-20-N of frozen concrete samples at 840 °D · D

Fig. 5.14 illustrates the results of compressive strength tests performed on A4 frozen concrete samples after

recovery curing. All frozen concrete samples displayed compressive strength values similar to A4-20-N after recovery curing, indicating that they did not suffer from early age frost damage. Moreover, it can be observed that pre-curing temperatures before early age freezing did not affect compressive strength at 840°D·D. The compressive strength of samples pre-cured at 20°C was approximately the same as that of samples pre-cured at  $5^{\circ}$ C.

Fig. 5.15 presents the compressive strength ratios based on A4-20-N of all frozen concrete samples at 840°C. As the compressive strength ratios were more excellent than 90%, it can be concluded that all frozen concrete samples were not damaged by early age frost. As per Fig. 5.5, the final setting time of A4 samples at 20°C was 6 hours and 13 minutes, while that at 5°C was 10 hours and 16 minutes. At 20°C, the A4 samples reached sufficient levels of strength and stiffness, indicating that they did not suffer from early age frost damage. In contrast, at 5°C, the initial setting time was 5 hours and 36 minutes, indicating that the cement in the concrete had not fully hydrated, and there was still much free water. When these samples were exposed to freezing at 6 hours (A4-5-F6 sample), the free water in the concrete froze, and the hydration reaction of the cement was interrupted. This can cause damage to the concrete and weaken its strength. However, subsequent curing of the frozen concrete under standard conditions resumed the hydration process and partially repaired the damage caused by early freezing.

Although the compressive strength values of frozen samples were lower than the required minimum compressive strength of 5.0 MPa when early age freezing occurred, the compressive strength ratios of frozen samples were greater than 90% after recovery curing, indicating that early age freezing did not affect frozen samples under A4 concrete conditions. This once again demonstrates that the strength development of concrete at early ages is not affected by freezing after the final setting time, which is consistent with the findings of Yamashita (2021) and Chapter 4 of this thesis.

Compared with Figs. 5.11 and 5.15, it can be concluded that, in the case of A4 concrete, the addition of 4 kg/m<sup>3</sup> of ACF additive is helpful in preventing early age frost damage as all samples can resist early age frost damage.



Fig. 5.14 Compressive strength of A4 frozen concrete samples after recovery curing



Fig. 5.15 Compressive strength ratios based on A4-20-N of frozen concrete samples at 840 °D · D

Fig. 5.16 presents the compressive strength results of frozen A6 concrete samples after recovery curing. All frozen concrete samples demonstrated the same compressive strength as A6-20-N after recovery curing, indicating that they were not affected by early age frost damage. Moreover, the pre-curing temperature before early age freezing did not affect the compressive strength at 840 °D ·D. The compressive strength of samples pre-cured at 20°C was approximately the same as that of samples pre-cured at 5°C.

Fig. 5.17 shows the compressive strength ratios of all frozen concrete samples based on A6-20-N at 840 °D.D. It can be observed that all frozen concrete samples resisted early age frost damage, as the compressive strength

ratios were above 90%. As per Fig. 5.5, the final setting time for A6 samples at 20°C was 5 hours and 38 minutes, while that at 5°C was 7 hours and 11 minutes. It indicates that A6 concrete samples cured at 20°C had reached adequate strength and stiffness within less than 6 hours and did not suffer from early age frost damage.

A6 samples had a final setting time of 7 hours and 11 minutes at 5°C, which suggests that the cement had almost completed its hydration reaction, forming a dense matrix of hydration products. It provided a robust skeleton structure for the concrete, enabling it to resist some of the pressure caused by the formation of ice lenses during freezing. When exposed to freezing at 6 hours (A6-5-F6 sample), the concrete withstood some of the pressure from the ice lenses due to its strong skeleton structure. Furthermore, it was observed that recovery curing under standard conditions for 31 days helped partially repair the damage caused by early freezing and restore the concrete's strength.

However, even though the compressive strength values of frozen samples were lower than the minimum required compressive strength of 5.0 MPa when early age freezing occurred, the compressive strength ratios of frozen samples were above 90% after recovery curing, indicating that early age freezing did not affect frozen samples under A6 concrete condition. This finding reinforces the earlier report by Yamashita (2021) and the results presented in Chapter 4 of this thesis that the strength development of concrete at early ages is not affected by freezing after the final setting time.

A comparison of Fig. 5.11 and Fig. 5.17 reveals that the addition of 6 kg/m<sup>3</sup> of ACF additive is helpful for preventing early age frost damage in A6 concrete, as all samples can resist early age frost damage.



Fig. 5.16 Compressive strength of A6 frozen concrete samples after recovery curing



Fig. 5.17 Compressive strength ratios based on A6-20-N of frozen concrete samples at 840 °D · D

Fig. 5.18 shows the relationship between the prevention effectiveness of early age frost damage and addition amounts of ACF additive under different temperatures. According to the results of compressive strength ratios based on each N sample, the following evaluation method for prevention effectiveness of early age frost damage is proposed using different addition amounts of ACF additive:

- (1) Compressive strength ratio below 90%: Damage due to freezing
- (2) Compressive strength ratio above 90%: No damage by freezing
- (3) Compressive strength ratio above 95%: No effect of freezing at all

It can be found that the prevention effectiveness between in Fig. 5.18 (a) P concrete samples and (b) A2 concrete samples conditions is nearly the same degree. It represents that the addition amount of 2 kg/m<sup>3</sup> can not improve the prevention effectiveness of early age frost damage. The F6 samples of P and A2 concrete in both 20°C temperature and 5°C temperature conditions were subjected to early age frost damage.

As for the conditions of Fig. 5.18 (c) A4 concrete samples and (d) A6 concrete samples, the compressive strength ratios of all samples are greater than 90%, which means that concrete samples were not subjected to early age frost damage. Especially in the case of F6 samples, the concrete did not suffer from early age frost damage. Compared with P concrete samples, the addition amount of 4 and 6 kg/m<sup>3</sup> can improve the prevention effectiveness of early age frost damage. In addition, A6 concrete is more effective than A4 concrete in preventing early age frost damage.



Fig. 5.18 Relationship between prevention effectiveness of early age frost damage and addition amounts of ACF additive under different temperatures

#### 5.5 Conclusion

In this chapter, the effect of shortening the setting time of concrete using different amounts of ACF additive and the effect on strength development under different amounts are investigated, and a suitable use program of ACF additive for preventing early age frost damage is discussed. The conclusions in Chapter 5 are given as follows:

(1) The use of ACF additive did not affect the slump and air content of fresh concrete.

(2) The addition of ACF additive resulted in a shorter setting time for concrete, and this effect was more pronounced at low temperatures. The greater the amount of ACF additive added, the shorter the setting time.

(3) The ACF additive can improve the early age strength within 24 hours. The early age strength at 7 days and compressive strength at 28 days of ACF concrete were the same as those of P concrete.

(4) Adding 4 and 6 kg/m<sup>3</sup> of ACF additive effectively prevented early age frost damage. These two addition amounts of ACF additive can be used for cold weather concreting due to their outstanding effectiveness in preventing early age frost damage.

To conclude, this study suggests that the addition of ACF additive is an effective method to prevent early age frost damage of concrete while maintaining its strength development. This study provides concrete producers and engineers with practical information to maximize the benefits of ACF additive in cold weather concreting.

## **References:**

RILEM recommendations for concreting in cold weather; VTT Technical Research Centre of Finland: Espoo, Finland, 1988.

Guide to Cold Weather Concreting; American Concrete Institute Committee 306; American Concrete Institute: Farmington Hills, MI, USA, 2016.

Karagol, F., Demirboga, and R., Khushefati, W.H., Behavior of fresh and hardened concretes with antifreeze admixtures in deep-freeze low temperatures and exterior winter conditions. Constr. Build. Mater. 2015, 76, 388–395.

Qin, X.C., Meng, S.P., Cao, D.F., Tu, Y.M., Sabourova, N., Grip, N., Ohlsson, U., Blanksvärd, T., Sas, G., and Elfgren, L., Evaluation of freeze-thaw damage on concrete material and prestressed concrete specimens. Constr. Build. Mater. 2016, 125, 892–904.

Cao, D.F., Qin, X.C., Meng, S.P., Tu, Y.M., Elfgren, L., Sabourova, N., Grip, N., Ohlsson, U., and Blanksvärd, T., Evaluation of prestress losses in prestressed concrete specimens subjected to freeze-thaw cycles. Struct. Infrastruct. Eng. 2016, 12, 159–170.

Zhang, G., Yu, H.; Li, H., and Yang, Y., Experimental study of deformation of early age concrete suffering from frost damage. Constr. Build. Mater. 2019, 215, 410-421.

Kim, M., Yokoyama, T., Tabata, M., Koh, Y., and Kamada, E., An experimental study on the effect of air entrainment for frost resistance of concrete at an early age. Archit. Inst. Jpn. 1978, 265, 1–10. (In Japanese)

Nmai, C.K., Cold weather concreting admixtures. Cem. Concr. Compos. 1988, 20, 121-128.

Yi, S., Pae, S., Kim, J., Minimum curing time prediction of early-age concrete to prevent frost damage. Constr. Build. Mater. 2011, 25, 1439–1449.

Cui, J., Duc Van, N., Zhang, F., and Hama, Y., Evaluation of Applicability of Minimum Required Compressive Strength for Cold Weather Concreting Based on Winter Meteorological Factors. Materials 2022, 15, 8490.

Hu, X., Peng, G., Niu, D., and Zhao, N. Damage study on service performance of early-age frozen concrete, Constr. Build. Mater. 2019, 210, 22–31.

Yamashita, K. Mechanism of Strength Enhancement Stagnation of Concrete Subjected to Freezing during the Setting and Hardening Process and Measurement of the Extent and Depth of Frost Damage. Ph.D. Thesis, Muroran Institute of Technology, Hokkaido, Japan, 2021. (In Japanese)

Ishii Y., Miyaguchi K., Urano S., and Yoda Y., Study on the Effect of Additives for Setting Time Adjustment in Cold Weather Concreting, Annual Journal of Japan Concrete Institute, Vol. 40, No. 1, pp. 231-236, 2018. (In Japanese)

Ishii Y., Ito S., and Araki A., Influence of Additives for Setting Time Adjustment on the Durability of Concrete, Annual Journal of Japan Concrete Institute, Vol. 41, No. 1, pp. 155-160, 2019. (In Japanese)

JIS A 1101, Method of Test for Slump of Concrete. Japan Standards Association: Tokyo, Japan, 2020. (In Japanese)

ISO 1920-2, Testing of Concrete-Part 2: Properties of Fresh Concrete. International Organization for Standardization: Geneva, Switzerland, 2016.

JIS A 1128, Method of Test for Air Concrete of Fresh Concrete by Pressure Method. Japan Standards Association: Tokyo, Japan, 2019. (In Japanese)

Standards Association: Tokyo, Japan, 2019. (In Japanese)

JIS A 1108; Method of Test for Compressive Strength of Concrete. Japan Standards Association: Tokyo, Japan, 2018. (In Japanese)

ISO 1920-4, Testing of Concrete-Part 4: Strength of Hardened Concrete International Organization for Standardization: Geneva, Switzerland, 2005.
## **CHAPTER 6**

## CONCLUSIONS

CHAPTER 6 CONCLUSIONS

#### 6.1 Introduction

In this study, the cold weather concreting guidelines in different countries are investigated to grasp the current situation of cold weather concreting. After summarizing the contents of the guidelines, a series of studies to develop technology for the diagnosis and prevention of early age frost damage are conducted. In addition, the frost resistance of concrete at early ages in the laboratory and outdoor exposure tests was studied.

# 6.2 Investigation of current situation of cold weather concreting aaccording to Guides to Cold Weather Concreting of various countries (Chapter 2)

This chapter has summarized eight specifications of cold weather concreting from various countries: Europe, the United States, Canada, Japan, China, South Korea, and Russia. Eleven content sections of cold weather concreting are selected, such as Application periods of cold weather concreting, Materials, Mix proportion design of concrete, Preparation for cold weather concreting, Curing methods, Early age curing, and Concrete quality management at job sites.

Most of the regulations of the Guides to Cold Weather Concreting of various countries are relatively similar, such as materials of concrete, curing methods, and preparation for cold weather concreting.

Each country has their unique construction methods and technologies, which may be related to various countries' climatic conditions and national standards.

The effect of the minimum required compressive strength values at early ages on frost resistance is still unclear. It is necessary to determine the applicability of minimum compressive strength for cold weather concreting.

With the development of new technologies, the diagnosis methods and prevention methods for early age frost damage also should be developed.

# 6.3 Verification of diagnostic method for early age frost damage depth by penetration tests (Chapter 3)

This chapter presents an experimental study to explore early age frost damage depth by penetration tests. It aims to develop accurate diagnostic methods for detecting the depth of early age frost damage. The penetration tests include the nail and pneumatic penetration test machine methods, and the penetration depth and diagnostic accuracy are discussed.

It is possible to diagnose whether the concrete is subjected to early age frost damage by a nail penetration test. But it is difficult to diagnose the depth of early age frost damage.

It can be seen that the penetration amount of the age 7 days has an excellent diagnosis effect for determining the early age frost damage and detecting the damage depth.

The penetration test by pneumatic penetration test machine is effective to diagnose the depth of early age frost damage on the age 7 days. It can roughly detect the damage depth with a micro- destructive degree.

# 6.4 Effect of compressive strength development at early ages on frost resistance of concrete (Chapter 4)

This chapter is divided into two parts to investigate the effect of compressive strength development at early ages on the frost resistance of concrete according to laboratory and outdoor exposure tests. The compressive strength test, hydration degree test, underwater weighing test, and freeze-thaw test were performed to discuss the influence. In addition, the applicability of minimum compressive strength for cold weather concreting is evaluated according to meteorological factors.

Air-entrained concrete that reaches a compressive strength of 5.0 MPa can withstand several freeze-thaw cycles and effectively prevent early age frost damage.

For early-age concrete subjected to repeated freeze-thaw cycles in a critical water-saturated environment, the frost resistance increases with the increase of compressive strength. For OPC concrete with w/c of 0.5, the numbers of freeze-thaw cycles of the concrete with compressive strength of 5.0, 12.0, 18.0, and 25.0 MPa that can maintain the relative dynamic modulus of elasticity above 90% were about 18, 55, 90, and 124 cycles, respectively.

Concrete that has finished its final setting can effectively resist early age frost damage not only in Airentrained condition but also in Non-air-entrained condition.

The frost resistance of concrete in outdoor exposure tests was lower than that of concrete in laboratory tests.

The dry-wet and freeze-thaw cycles in the natural environment can reduce the frost resistance of concrete.

# 6.5 Investigation of the effect of preventing early age frost damage using additive for setting time adjustment (Chapter 5)

In order to develop an effective prevention method for early age frost damage using additive for setting time adjustment, the effect of shortening the setting time of concrete at room temperature and low temperature is investigated, the effect on strength development under different amounts is discussed, and a suitable use program of ACF additive based on the prevention effectiveness of early age frost damage is proposed.

The use of ACF additive did not affect the slump and air content of fresh concrete.

The addition of ACF additive resulted in a shorter setting time for concrete, and this effect was more pronounced at low temperatures. The greater the amount of ACF additive added, the shorter the setting time.

The ACF additive can improve the early age strength within 24 hours. The early age strength at 7 days and compressive strength at 28 days of ACF concrete were the same as those of P concrete.

Adding 4 and 6 kg/m<sup>3</sup> of ACF additive effectively prevented early age frost damage. These two addition amounts of ACF additive can be used for cold weather concreting due to their outstanding effectiveness in preventing early age frost damage.

#### 6.6 Summary and future work

Considering the construction quality of cold weather concreting, the prevention and diagnosis of early age frost damage have always been important to the construction. Based on the contents of the current cold weather concreting guidelines in various countries, this study investigated the frost resistance of concrete at early ages. From the standpoint of new technology development, this study tried a new diagnostic method, and discussed the effects of preventing early age frost damage using the additive for setting time adjustment. The results obtained in this study provide new ideas and methods for diagnosing and preventing early age frost damage for cold weather concreting.

The results show that the diagnostic method of penetration tests has high accuracy in detecting the depth of

early age frost damage, which may be applied to the construction site. The additive for setting time adjustment can shorten the setting time of concrete even at low temperatures, which improves the effect of preventing early age frost damage. It can be used for cold weather concreting and improve concrete quality.

**ACKNOWLEDGEMENTS** 

#### **ACKNOWLEDGEMENTS**

Studying at Muroran Institute of Technology was an excellent experience in my life that taught me a lot. The experience not only gave me knowledge, but also gave me a group of friends with dreams and power, as well as a goal to strive for in life. I would like to express my sincere gratitude to all the kind people around me for helping and supporting me in completing my doctoral dissertation.

First of all, I would like to thank my supervisor, Professor Hama Yukio, for his teaching, and I am grateful to him. His academic style of rigorous knowledge and excellence is my guiding light on the scientific road. His modest, elegant, warm, and friendly personality are the realm of life I have been striving for. Writing the thesis was not easy, from the initial conception of the topic, experimental design, data collection, and collation to the final draft, each step was inseparable from Professor Hama Yukio's guidance and teaching. Whenever I encountered a bottleneck in my experiments, his high-level guidance gave me hope and strength to move forward, just like setting the clouds into the sun. Professor Hama Yukio's professional counseling, patient guidance in methodology, and strict academic teaching have helped me to know how to find and solve problems scientifically as a Ph.D. student and to learn how to be a qualified researcher and experience the fun of exploration.

I would like to express my sincere thanks to Professor Mizoguchi and Associate Professor Takase, my vice supervisors, for their valuable advice on my doctoral dissertation revision and doctoral dissertation presentation.

Furthermore, I would like to express my gratitude to Associate Professor Jihoon Kim. He always encourages me and provides kind recommendations for my research, which is crucial to finishing a significant portion of my dissertation.

Special thanks are extended to post-doctor Nguyen Duc Van, who gave me some valuable advice regarding my academic writing and gave me helpful advice for my studies. I greatly appreciate his assistance and practical suggestions for my journal papers.

In addition, I would like to express my deep appreciation to my friends, Dr. Noguchi, Dr. Ding, Mr. Fukuda, Mr. Zhang, Ms. Luo, Mr. Yamada, Mr. Takahashi, Mr. Kojima, and so on. I sincerely appreciate their support in my life and my research. My doctoral study journey became colorful and unforgettable because of their friendship, kindness, and encouragement during my doctoral studies abroad.

**ACKNOWLEDGEMENTS** 

I would like to thank my parents very much. They were always the first to encourage me whenever I encountered difficulties. Looking back on the road I have traveled, every footprint is filled with their selfless love and wise advice. The five-year road to study abroad is full of earnest expectations for me. Their selfless support, both spiritual and material, has strengthened my belief in pursuing my dream in life.

I am very grateful to my sister, Yujie Cui, who always worried about my daily life and took care of me in every way during my first three years in Japan. While completing her Ph.D. study, my sister encouraged me to finish my master's and Ph.D. studies by teaching me through her words and example. I also want to express my gratitude to my sister's husband, Weichen Liu, who always encourage me to insist on my study. I have learned a lot from them. They gave me a lot of instruction and support. Their extraordinary characteristics have impacted and altered my life. Thank you all so much!

Finally, I would like to thank my wife, Sun Xueting. She has always been there for me during my doctoral studies, encouraging me to overcome difficulties and always watching over me. She always supported and respected my ideas and gave me a lot of help. I can't describe how grateful I am to my wife, and I want to spend my whole life taking care of her and caring for her. Thanks for the love my wife has shown me all the way. I love you!

### LIST OF PAPERS

#### Publication:

Jiahui Cui,Nguyen Duc Van, Feng Zhang, Yukio Hama, Evaluation of Applicability of Minimum Required Compressive Strength for Cold Weather Concreting Based on Winter Meteorological Factors, Materials, 2022, 15, 8490.

### **Presentation:**

Jiahui CUI, Nguyen Duc VAN, Jihoon KIM, Yukio HAMA, Literature Review of Guides to Cold Weather Concreting of Various Countrie, Proceedings of 14th International Symposium between Japan, China and Korea Performance Improvement of Concrete for Long Life Span Structure, 2021.8, pp. 169-176.