

Doctoral Dissertation

Mechanical Properties and Durability  
Performance of Concrete Subjected to High  
Temperature Heating

A Dissertation Submitted to Tokushima University in Partial Fulfillment of  
the Requirements for the Doctor's Degree

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## Chapter 1 Introduction

### 1.1 Background

Concrete is the most widely used construction material in civil engineering due to its excellent mechanical and durability performance and the low cost since it was firstly developed in 19th century. As the progress of material science in the recent years, more and more new types of concrete such as self-compacting concrete, high-strength concrete, high-performance concrete, recycled concrete, light-weight concrete, porous concrete and fiber reinforced concrete, etc. were developed and applied in the actual engineering projects. In order to accelerate the application of the concrete in civil engineering, a lot of researchers conducted a series of experiments and simulations to investigate the properties of concrete such as the compressive strength, tensile strength, elastic modulus, stress-strain relationship, cracking, expansion, porosity, alkali-silica reaction, ion penetration resistance, steel corrosion and so on.

Since concrete is a kind of long-term used construction material which can be used as long as 50 years and more, the long-term properties of concrete such as the fire resistance and durability performance are need to be valued. Both the mechanical and durability performance of the concrete will be severely deteriorated due to the destruction of the concrete micro-structure when the concrete is subjected to a high temperature environment. After fire, the severely deteriorated concrete cannot be used anymore and will be replaced using the new concrete in the RC (reinforced concrete) structures. However, the concrete is still can be used after some strengthening and rehabilitation if the heating temperature is not too high. On the other hand, the residual durability properties of the fire damaged concrete will also be influenced by the high temperature even the deterioration of the mechanical properties is not serious.

#### 1.1.1 Fire resistance of concrete

Fire is a kind of frequently occurred disaster which can cause huge losses [1]. The frequency of fire accident is the highest among other disasters according to the World Fire Statistics Centre (WFSC) of United Nations. There are about 6~7 million fire accidents around the world every year and about 65000~75000 people are dead in these disasters. The direct fire losses and fire deaths of some countries are shown in Table 1.1 and

Table 1.2 respectively [2].

Table 1.1 Direct fire losses and as average percentage of GDP.  
(Unit: millions, except for Japan-billions)

Country	Currency	Direct Losses			Percentage of GDP (2008-2010)
		2008	2009	2010	
Hungary	Ft		580	210	0.02 [2009-2010]
Singapore	\$S	110	115	115	0.04
Slovenia	SIT				0.07 [2002-2004]
Australia	\$AUS	1,000	955	940	0.07
Czech	Kč	3,700	2,450	2,200	0.07
Spain	€	910			0.08 [2008]
Poland	zł	1,450	1,150		0.09 [2007-2009]
US	\$US	17,500	14,000	13,000	0.10
Japan	¥	615	610	565	0.12
NZL	\$NZ	240		210	0.12
Germany	€	2,850	2,950	2,700	0.12
UK	£	1,950	1,750	1,750	0.13
Netherlands	€	1050	925	675	0.15
Finland	€	305	280	330	0.17
Sweden	kr	5,950	5,550	5,650	0.18
Denmark	kr				0.20 [2005-2007]
France	€	4,550			0.20
Italy	€	3,150	3,750	2,600	0.20
Norway	kr				0.22 [2003-2005]

Normally, the mechanical properties of concrete would be severely damaged due to the thermal stress and the high vapor pressure when the concrete structure is subjected to a high temperature environment [3-6]. Such damages will become serious and the concrete can't be used anymore when the temperature exceeds 500 °C (the residual compressive strength will decrease to less than 30% of the un-heated concrete) especially for the high-density concrete such as the self-compacting concrete, high performance concrete, high-strength concrete and so on. However, in the cases of the concrete subjected to the heating below 500 °C, the high temperature damaged concrete could be generally used after the rehabilitation and strengthening, although the durability performance of

such concrete members has not been clarified enough.

Table 1.2 Fire deaths and population comparisons.

(Additions refer to the fire deaths unknown to the fire brigades or not recorded on death certificates.)

Country	Addition (%)	Fire Deaths			Deaths Per 100,000 Population (2008-2010)
		2008	2009	2010	
Singapore	10	1	1	1	0.02
Switzerland	15	30	25	25	0.34
Italy	25	285	285	240	0.45
Netherlands	5	100	60	70	0.46
Austria	5	55	40		0.47 [2007-2009]
Slovenia	5	10	10	10	0.49
Spain	25	270	205	235	0.52
Portugal	Nil.	65	55	60	0.57
Germany	25	500	540	465	0.60
Australia	Nil.	120	270	90	0.73
UK	5	475	460	445	0.60
Canada	10	295	240		0.77 [2007-2009]
NZL	Nil.	35	40	25	0.77
France	25	595	595		0.96 [2007-2009]
Greece	25	130	110	110	1.05
US	6.4	3,650	3,300	3,400	1.11
Norway	0.5	70	55	40	1.14
Ireland	25	45	55	55	1.17
Belgium	25				1.21 [2004]
Czech	10	150	130	145	1.35
Denmark	Nil.	90	70	65	1.36
Sweden	12.5	130	140	145	1.49
Japan	2	2,000	1,950	1,800	1.51
Poland	5	585	565	595	1.52
Hungary	Nil.	180	140	140	1.53
Barbados	Nil.	5			1.65 [2007-2008]
Romania	Nil.	410	355	395	1.76
Finland	5	110	120	95	2.03

### 1.1.2 Durability performance of concrete

In terms of durability performance of concrete, chloride-induced corrosion of the steel bars embedded in concrete is the main cause of the deterioration of reinforced concrete (RC) members especially when structures are located in a marine environment [7-9]. According to the conservative estimates, in the developed countries one-half of highway bridges are deteriorating due to the corrosion of reinforcement, and billions of dollars are required to repair or rehabilitate the damaged structures. Table 1.3 lists some cases of damage associated with corrosion of steel in concrete as well as the economic loss caused by damage [10].

Table 1.3 Damage and economic loss associated with corrosion of steel embedded in concrete.

Event	Damage	Economic Loss
Estimate in USA/1991	Corrosion damage of highway bridges	\$90~150 billion
Estimate in USA/1991	Annual cost of repairs of bridge deck, substructures and car parks	\$200~450 million
Estimate in UK/1989	Corrosion damage of motorway and trunk road bridges in England and Wales	GBP616.5 million
Estimate in UK/1989	Annual cost of repairs to concrete structures	GBP500 million
Collapse of the Berlin Congress hall/1982	Collapse	
Collapse of multistory parking structure in Minnesota/1986	Collapse	
Collapse of post-tensioned concrete bridge in Wales/1988	Collapse	
Slab spalled off a bridge in New York/1997	One man killed	

In general, the porosity, cracking situation and chloride ion (Cl<sup>-</sup>) penetration resistance of concrete are all main factors that affect the steel corrosion in concrete. These durability properties might be severely

damaged by the high temperature even the deterioration of mechanical properties is not significant when the heating temperature is not too high. In order to rehabilitate and strengthen the fire damaged concrete structures, such durability properties concerning to the steel corrosion in concrete must be investigated and valued.

### 1.1.3 Self-compacting concrete and copper slag

Compared with the normal concrete, self-compacting concrete has a weaker fire resistance performance although it has been widely used in actual applications due to its improved rheological properties and durability enhancement [11-14] since it was first developed in Japan in the late 1980s [15]. Normally, the self-compacting concrete has a higher density compared with the normal concrete, which will generate the higher vapor pressure caused by the evaporation of the water in concrete when the concrete is subjected to the high temperature environment. This high vapor pressure can cause the explosive spalling of the concrete and severely destroy the micro-structure of the concrete.

According to the previous related researches, different kinds of additives such as fibers and recycled aggregates mixed in the self-compacting concrete can improve its fluidity and fire resistance properties. However, the using of additives always deteriorates the mechanical and durability performance of the self-compacting concrete. In addition, the influences of different types of the additives on the mechanical and durability properties of concrete are also different. In order to accelerate the application of the different additives in self-compacting concrete, the related researches for the basic properties of self-compacting concrete containing different kinds of additives must be carried out.

On the other hand, copper slag (CUS) is a by-product generated during the process of copper smelting [16]. Production of one ton of copper produces about three tons of copper slag and about 24.6 million tons of copper slag is generated from world copper production every year [17]. The recycled copper slag can be used in concrete as a substitute for fine aggregate and improve the fluidity of the concrete [18-22], while the mixing of the copper slag fine aggregate also would promote the bleeding of the fresh concrete due to the high density and glassy smooth surface of the copper slag [23-24]. In order to reduce the bleeding of the concrete containing copper slag fine aggregate, the application of copper slag fine

aggregate to powder rich concrete like self-compacting concrete might be effective.

Although mixing copper slag aggregates into self-compacting concrete could both improve the fluidity of fresh concrete and reduce the bleeding caused by the copper slag fine aggregate, the residual mechanical and durability properties of high temperature damaged self-compacting concrete containing copper slag fine aggregate are still unknown and it has a great significance to carry out such related researches in order to investigate its properties and accelerate the application of copper slag as a substitute for fine aggregate in concrete.

## 1.2 Research purpose and objective

In general, the severely deteriorated concrete during a fire can't be used anymore and will be replaced by the new concrete. However, the slightly deteriorated concrete by fire still can be used after strengthening and rehabilitation if the heating temperature is not too high. Although the residual mechanical properties of high temperature damaged concrete fill the requirements of the constructions, the long-term durability properties of the high temperature damaged concrete such as chloride ion penetration resistance and steel bar corrosion resistance properties are still not clarified enough especially for the different kinds of new type concrete such as self-compacting concrete, high-strength concrete, high-performance concrete and recycled concrete etc.

In order to investigate the residual mechanical and durability properties of high temperature damaged concrete and offer data support for the strengthening and rehabilitation of fire damaged concrete constructions. Normal concrete with different water to cement ratios (W/C) and self-compacting concrete containing copper slag fine aggregate were prepared and conducted with high temperature experiment in this dissertation. The residual mechanical properties like compressive strength, splitting tensile strength, mass loss, porosity, elastic modulus, stress-strain curves and the residual durability properties like chloride ion penetration and steel bar corrosion of the high temperature damaged concrete were measured and comparatively analyzed. In addition, the micro-structure of the high temperature damaged concrete was also observed by using scanning electron microscope (SEM).

Based on the experimental results and comparative analysis, the following objectives were completed in this dissertation:

(1) The influence of high temperature on residual compressive strength of normal concrete and self-compacting concrete was clarified.

(2) The chloride ion distribution and penetration pattern in high temperature damaged concrete are obtained.

(3) The chloride-induced steel bar corrosion situation including half-cell potential, polarization resistance, concrete resistivity and steel bar weight loss in high temperature damaged concrete was measured.

(4) The influence of recycled copper slag as a substitute for fine aggregate in concrete on mechanical and durability properties of high temperature damaged self-compacting concrete was obtained.

(5) The residual stress-strain curves of high temperature damaged self-compacting concrete containing copper slag fine aggregate were measured.

(6) The relationship between porosity and the heating temperature of normal concrete and self-compacting concrete were measured.

(7) The micro-structure of high temperature damaged concrete was observed by using SEM.

### 1.3 Dissertation organization

In this dissertation, totally five chapters were written and the main contents of the five chapters are listed as follows:

Chapter 1 introduced the background, purpose and objectives of the research.

Chapter 2 mainly summarized the previous researches related to the mechanical and durability performances of the high temperature damaged normal concrete, self-compacting concrete and concrete containing different kinds of additives and recycled aggregate.

Chapter 3 is the experimental research on the residual mechanical properties, chloride induced steel bar corrosion and chloride ions distribution situation of normal concrete with different W/C (water to cement) ratios after heating up to 300 °C.

Chapter 4 is the experimental research on the residual mechanical properties and chloride ions penetration resistance performance of self-compacting concrete containing copper slag fine aggregate after

heating up to 400 °C.

Chapter 5 is the conclusions of this research and the directions of the future works.

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## Chapter 2 Previous researches related to the properties of concrete damaged by high temperature

Concrete is the most widely used construction material in civil engineering since it was firstly developed in 19th century and fire is a kind of frequently occurred disaster which can cause huge losses. The mechanical and durability properties of concrete will be severely deteriorated by the high temperature and a lot of researchers have investigated the influence of high temperature on the residual mechanical and durability properties of fire damaged concrete.

### 2.1 Mechanical properties

#### 2.1.1 Compressive strength

One of the most important properties of concrete is the compressive strength. It has been reported that [1-5] the residual compressive strength of concrete will decrease with the increase of heating temperature and the concrete will lose more than 70% of its compressive strength if the heating temperature exceeds 500 °C [6].

Basically, the aggregates and cement paste in concrete will expand to different degrees when the concrete is subjected to a high temperature environment due to the different thermal expansion rates of the aggregates and cement paste in concrete. The different expansions of the aggregates and cement paste will create micro-cracks in concrete and the developing of these micro-cracks will become serious as the heating temperature increases, which will eventually cause the deterioration of the compressive strength of the fire damaged concrete [7].

On the other hand, the high-density concrete such as self-compacting concrete, high-strength concrete and high-performance concrete has a weaker fire resistance performance compared with the case of normal concrete. The reason is considered that the steam generated by high temperature in concrete is difficult to release due to the high density of the concrete. The steam in high density concrete will generate higher vapor pressure and easier to occur explosive spalling compared with that of the normal concrete. In order to prevent the explosive spalling, various kinds of fibers and additives are mixed in concrete. However, these additives also will reduce the compressive strength of the concrete [8-11]. In addition,

recycled aggregates such as metal slag, foundry sand and glass used in recycled concrete also will decrease the residual compressive strength of fire damaged concrete [12-15] due to the developing of micro-cracks caused by the different thermal expansion of the recycled aggregates and cement paste.

However, some researchers also reported that the residual compressive strength of high temperature damaged concrete doesn't decrease and even increase if the heating temperature is not too high [1, 16-18]. Although the precise explanation has not been specified, it is considered due to the re-hydration in concrete caused by the hot steam curing effect [9, 19-20].

### 2.1.2 Explosive spalling



Figure 2.1 Explosive spalling of reinforced concrete columns.

Explosive spalling of concrete is a severe threat to the RC structure when fire occurs. Once the concrete spalled during a fire, the steel bars embedded in concrete will expose to high temperature directly, which will accelerate

the deterioration of the RC structure [21]. The explosive spalling situations of reinforced concrete columns are shown in Figure 2.1.

In general, the explosive spalling is considered to be caused by the combining effect of vapor stress and thermal stress [22]. The high density concrete such as self-compacting concrete, high-strength concrete and high performance concrete will generate higher vapor pressure when the concrete is subjected to a high temperature environment because the steam in high density concrete is difficult to release, which means that the explosive spalling susceptibility of high density concrete is higher than the case of normal concrete [23].

C. Zhang et al. [24] found that the explosive spalling was occurred at 370 °C for the concrete which compressive strength is 60 MPa. K.K. Sideris [25] found that the concrete which compressive strength is 50 MPa occurred explosive spalling from 380 °C to 450 °C, whereas in the case of the concrete which compressive strength is 30 MPa, explosive spalling was occurred from 500 °C to 580 °C. Many researchers conducted high temperature experiments on concrete [26-28] and reported that normally the concrete explosive spalling temperature decreases with the increase of compressive strength and all specimens were exploded when the heating temperature exceeds 800 °C despite the compressive strength.

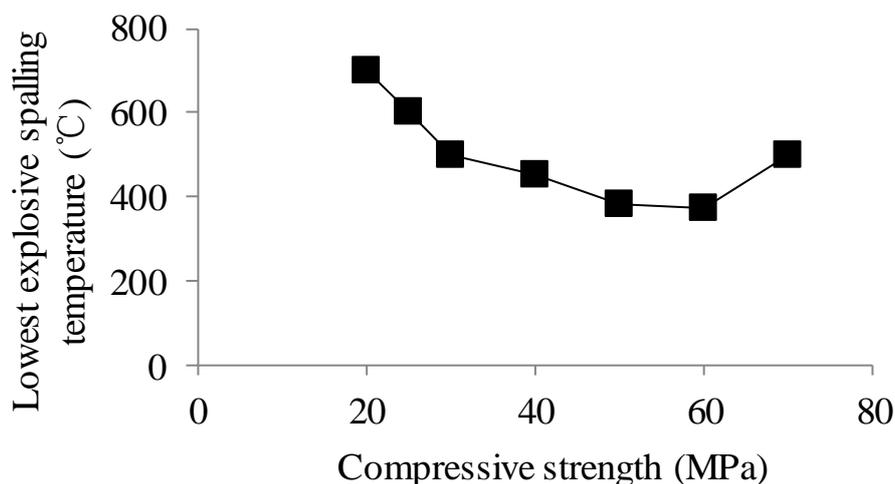


Figure 2.2 Relationship between the lowest explosive spalling temperature and compressive strength of self-compacting concrete.

The relationship between the lowest explosive spalling temperature and compressive strength of the self-compacting concrete is shown in Figure

2.2 [29]. It can be seen from Figure 2.2 that the lowest explosive spalling temperature of self-compacting concrete decreases with the increase of compressive strength when the compressive strength is lower than 60 MPa. However, the lowest explosive spalling temperature increases with the increase of compressive strength after that.

Mixing various kinds of fibres and additives into concrete is an effective method to prevent explosive spalling when the concrete is subjected to high temperature [30]. Y. Ding et al. [27] found that mixing polypropylene (PP) fibre into concrete can decrease the susceptibility of explosive spalling whereas the influence of steel fibre on explosive spalling is not obvious. K.K. Sideris [11] also found that mixing PP fibre can improve the explosive spalling resistance of concrete but also decreases the compressive strength of the concrete. G. Mathew et al. and M.F. Nuruddin et al. reported that [31-32] adding fly ash into concrete also can decrease the susceptibility of explosive spalling.

A. Noumowe et al. [33] found that temperature rising rate and loading situation can not affect the explosive spalling temperature but the spalling degree is increased with the increase of loading. It can be obtained from above researches that explosive spalling of concrete is a complex phenomenon affected by many factors including temperature, compressive strength, porosity, and loading situation et al.

### 2.1.3 Micro-structures

The micro-structure is an important factor to assess the fire resistance and durability performance of concrete. The aggregates and cement paste will expand to different degrees due to its different thermal expansion rates and generate micro-cracks when the concrete is subjected to a high temperature environment. The micro-cracks caused by high temperature is shown in Figure 2.3.

The development of micro-cracks in concrete can release the vapor pressure caused by the evaporation of water in concrete under high temperature and decrease the susceptibility of explosive spalling. However, the expansion of micro-cracks and voids also deteriorates the ion penetration resistance performance of the high temperature damaged concrete, which will eventually cause the steel bar corrosion in concrete [34].

Related researchers found that [35-36] the residual porosity of fire

damaged concrete is increased with the increase of heating temperature. From 20 °C to 200 °C, the porosity increases mainly due to the evaporation of free water in the capillary in concrete. From 200 °C to 500 °C, the increase of porosity is relatively small due to the evaporation of chemical bounding water in concrete. After 500 °C, the residual porosity of concrete significantly decreases with the deterioration of the mechanical and durability properties of concrete mainly caused by the different thermal expansion of the aggregates and cement paste.

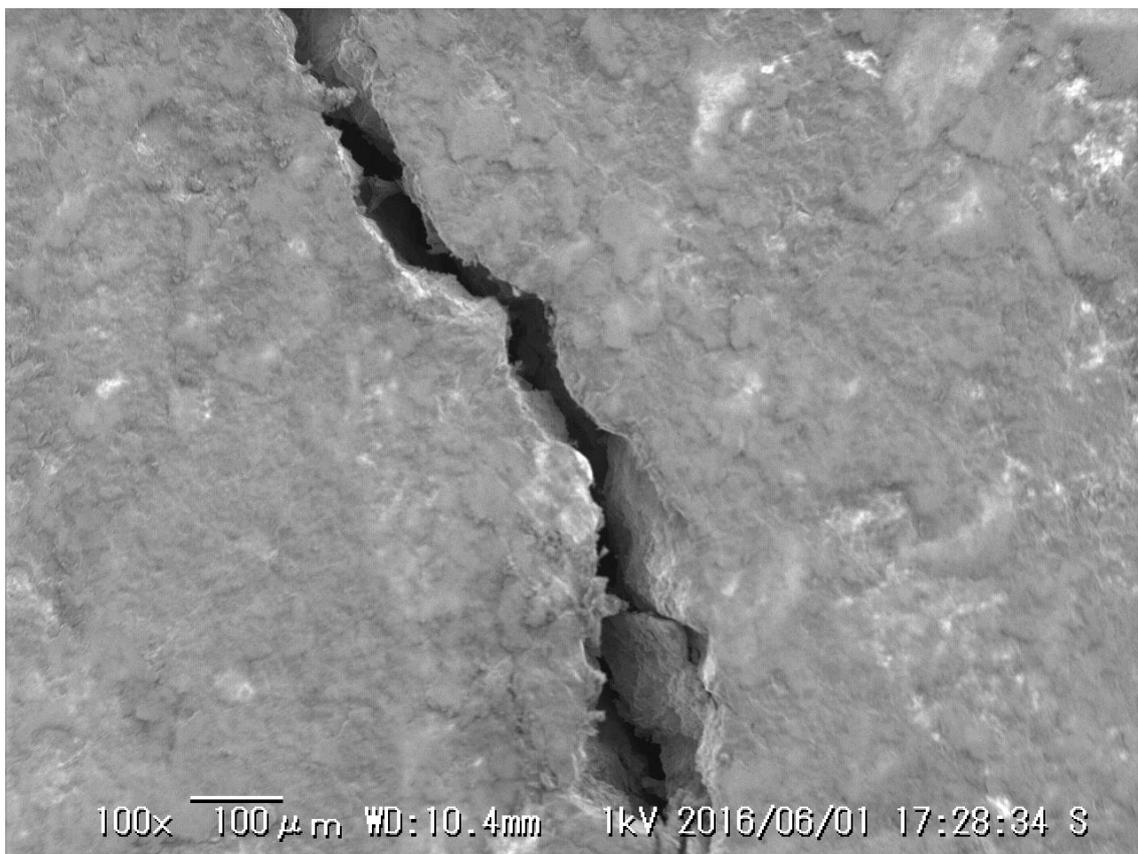


Figure 2.3 Micro-crack caused by high temperature in concrete.

PP fibre is often used in concrete in order to prevent explosive spalling especially for the high-density concrete, which will melt in a high temperature environment and generate small channels in concrete. The small channels can release the high vapor pressure effectively but also reduces the residual mechanical performance of the high temperature damaged concrete [37].

The use of various kinds of recycled aggregates in concrete such as metal slag, foundry sand and glass will also affect the micro-structure of

concrete. C.Q. Lye et al. found that [38] the mixing of copper slag aggregate into concrete can promote the bleeding of the fresh concrete due to its high density and glassy smooth surface. The bleeding water will generate micro-cracks and voids in concrete and deteriorate the compressive strength of the concrete. In addition, the different thermal expansion rates between the recycled aggregates and cement paste will also promote the developing of micro-cracks.

T.C. Ling, et al. also found that [13] the water porosity of high temperature damaged recycled glass concrete is decreased with the increase of the content of recycled glass aggregate. The reason should be the pore-filling effect of the molten recycled glass-that the recycled glass aggregate will melt and fill the pore voids under high temperature, which will eventually reduce the total water porosity in the concrete upon re-solidification.

## 2.2 Durability properties

The durability performance is an important factor to assess the long-term properties of RC structures. Fire damaged concrete will generate many micro-cracks inside even its mechanical deterioration is not obvious when the heating temperature is not too high. The development and expansion of these micro-cracks will destroy the integrity of concrete cover, which will lead to the invasion of different kinds of ions and eventually cause the steel bar corrosion.

### 2.2.1 Ion penetration

Since the corrosion of steel bars embedded in concrete is the main cause of mechanical deterioration of RC members and the steel corrosion is mainly caused by the  $\text{Cl}^-$  penetrated into the concrete, the ion penetration resistance performance of concrete is an important factor to assess the durability of the concrete.

Some researchers have reported that [39-40] the damage caused by the high temperature can accelerate the chloride permeability of concrete and the acceleration rate increases with the increase of the heating temperature. Generally,  $\text{Cl}^-$  ions penetrate from the concrete surface as the apparent diffusion process with the fixing of a part of free  $\text{Cl}^-$  to the cement hydration products. However, in the cases of concrete heated to around 200 °C or 300 °C, the penetration of  $\text{Cl}^-$  seems different from the usual diffusion

process observed in the general cases [41] and such  $\text{Cl}^-$  profiles will promote the steel corrosion in concrete.

In the cases of high density concrete, normally its ion penetration resistance performance is better than that of the normal concrete under ambient temperature. However, the high temperature damaged deterioration degree of the high density concrete is also severer than the case of normal concrete, which means that the high temperature damaged high density concrete has a weaker ion penetration performance compared with that of the normal concrete [42]. In addition, as for the high density concrete containing PP fibre subjected to high temperature, the channels formed by the melting of PP fibre also will accelerate the penetration of  $\text{Cl}^-$  in to concrete [43-44].

Previous researches [45-46] related to the recycled concrete containing different kinds of recycled aggregates show that the high density and glassy smooth surfaced recycled aggregates can promote the bleeding of fresh concrete, the bleeding water will generate a lot of micro-cracks and voids around the interfaces between the recycled aggregates and cement paste. These initial defects will deteriorate the ion penetration resistance performance of concrete especially when the micro-cracks and voids are developed and expanded during the heating. On the other hand, some kinds of recycled aggregates like glass will melt and fill in the micro-cracks and voids under high temperature which can improve the residual ion penetration resistance performance of the fire damaged concrete.

### 2.2.2 Steel corrosion

Corrosion of reinforcing steel bars and other embedded metals in concrete is the leading cause of deterioration of the mechanical properties of reinforced concrete members especially when the structure is located in a marine environment. When the steel bars corrode, the resulting rust will occupy a greater volume than the steel bars and cause expansion. This expansion creates tensile stresses in the concrete, which can eventually cause concrete cracking and spalling [47].

Previous related researches [48-49] concerning to the steel corrosion of high temperature damaged concrete show that the concrete micro-structure will be destroyed by high temperature and the chloride ions can penetrate the concrete more easily, the steel bar embedded in the fire damaged concrete is more prone to be corroded than the case of non-heated concrete.

Basically, the steel corrosion in concrete depends on the Cl<sup>-</sup> situation of the concrete. Although many researchers have investigated the ion penetration resistance performance of high temperature damaged concrete as shown in 2.2.1, there is almost no research directly concerning to the steel corrosion situation of fire damaged RC structure.

### 2.3 Summary

From the previous related researches, the following conclusions can be summarized:

(1) The residual mechanical properties of fire damaged concrete will be severely deteriorated when the heating temperature exceeds 500 °C. However, the residual compressive strength of fire damaged concrete will not decrease and even increase due to the re-hydration of cement paste caused by the steam curing effect if the heating temperature is not too high.

(2) The development and expansion of the micro-cracks in concrete caused by high temperature will destroy the micro-structure of concrete even the deterioration of residual mechanical properties may not obvious if the heating temperature is not too high, which will make the ions penetrate into the high temperature damaged concrete more easily.

(3) Basically, the high density concrete likes self-compacting concrete, high-strength concrete and high performance concrete has a weaker fire resistance performance compared with the normal concrete. The steam generated in high density concrete is difficult to be released and will cause a higher vapor pressure than that of the normal concrete, which will eventually lead to the occurrence of explosive spalling.

(4) Mixing of various kinds of additives such as PP fibre and steel fibre into concrete can improve the fire resistance performance of the concrete and reduce the susceptibility of explosive spalling, but the using of these additives also deteriorates the residual mechanical properties of the high temperature damaged concrete.

(5) The influences of different kinds of recycled aggregates on the fire resistance performance of concrete are also different and the residual properties of fire damaged recycled concrete are still not be clarified enough. In order to accelerate the application of recycled aggregates in concrete, the fundamental researches related to the fire resistance performances of concrete containing various kinds of recycled aggregates

are still need to be studied in the future.

(6) Although many researchers investigated the micro-structure deterioration and  $\text{Cl}^-$  penetration patterns of high temperature damaged concrete, the direct steel corrosion situation in fire damaged concrete is still not clear.

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## Chapter 3 Experimental research on the properties of normal concrete after heating up to 300 °C

In this chapter, the chloride-induced corrosion of steel in concrete subjected to the heating up to 300 °C was investigated. Reinforced concrete specimens were prepared for the heating followed by the cyclic immersion into salt water and drying for accelerating steel corrosion. Electrochemical corrosion indexes were measured during the acceleration period. After the accelerated steel corrosion test, the distributions of chloride ions ( $\text{Cl}^-$ ) in concrete and the weight loss of the steel bar in concrete were measured. Moreover, micro-structure of the concrete was observed by SEM (Scanning Electron Microscope) observation.

### 3.1 Materials and specimens

#### 3.1.1 Mixture proportions

Mixture proportions of concrete used in this program are shown in Table 3.1. Water to cement ratios (W/C) were set to 45% and 60% respectively. Ordinary portland cement (density: 3.16 g/cm<sup>3</sup>, R2O: 0.56%), fine aggregate (density: 2.57 g/cm<sup>3</sup>) and coarse aggregate (density: 2.57 g/cm<sup>3</sup>) were used in this program, the volume rate of sand to total aggregate (s/a) was 48% and the maximum size of coarse aggregate ( $G_{\text{max}}$ ) was 15 mm. Water reducing agent (WRA) and Air entraining agent (AEA) were also used in this program to satisfy the requirements of the fresh concrete. The data of slump and concrete air were shown in Table 3.1.

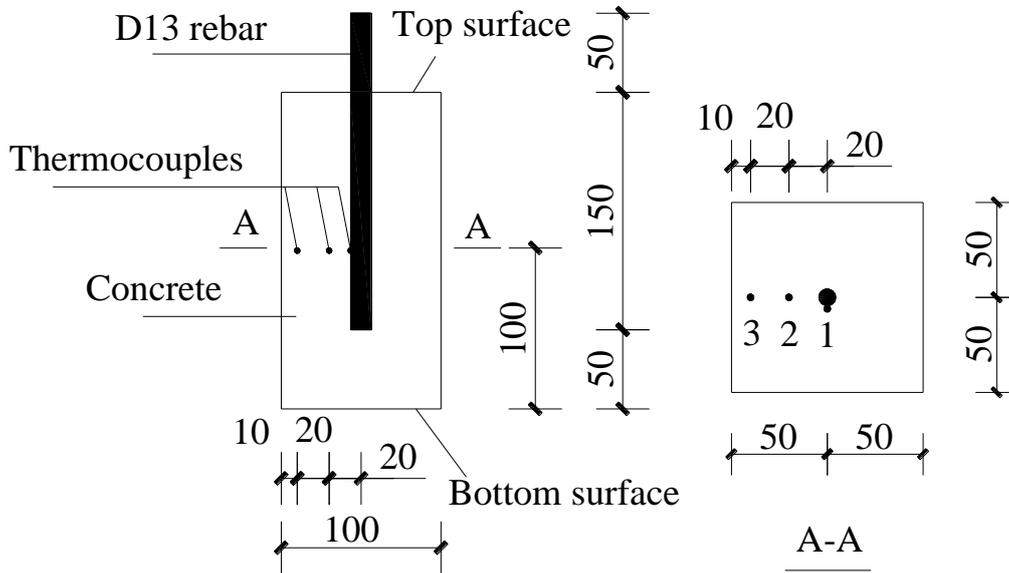
Table 3.1 Mixture proportions of concrete.

W/C (%)	s/a (%)	Slump (cm)	Air (%)	Content (kg/m <sup>3</sup> )					
				C	W	S	G	WRA	AEA
45	48	3.5	2.3	389	175	804	871	1.17	0.0125
60		9.0	2.0	300	180	833	903	0.50	0.0095

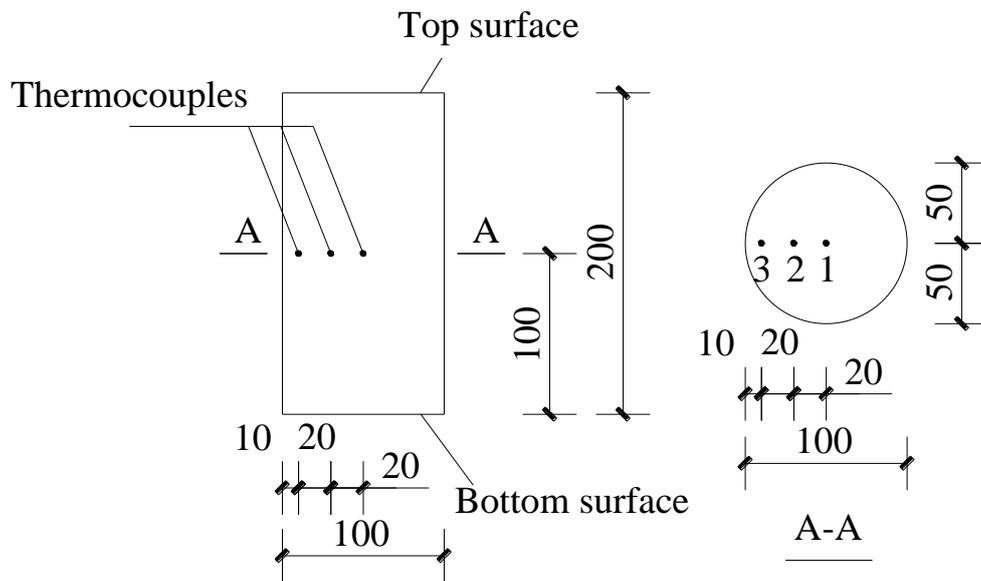
#### 3.1.2 Preparation of specimens

Two kinds of specimens were prepared in this program and the details are shown in Figure 3.1. The prism specimens with D13 (deformed steel bar which diameter is 13 mm) rebar were prepared to investigate the chloride-induced steel corrosion in concrete after the heating test and cylinder specimens without rebar were prepared to measure the residual

compressive strength after the heating.



(a) Prism specimens



(b) Cylinder specimens

Figure 3.1 Outlines of specimens. (Unit: mm)

Three K-H type thermocouples which can tolerate the high temperature up to 300 °C were embedded in one specimen of each group in order to measure the temperature in concrete during the heating. The locations of thermocouples in concrete are also shown in Figure 3.1 and the thermocouples were fixed in the molds during the casting process using the

ion wire. In order to eliminate the influence of ion wire on the thermocouples during the heating test, the temperature sensing point of the thermocouples were set to separate from the iron wire and the iron wire were covered with thick plastic cover. The thermocouples used in this program are shown in Figure 3.2.



Figure 3.2 Thermocouples used in the experiment.

All specimens were demolded 24 hours after the specimens were casted. Then the specimens were covered with wet cloth, plastic wrap and tape. At last, the specimens were cured in a room controlled constantly at 20 °C for 28 days before heating as shown in Figure 3.3.



Figure 3.3 Specimens curing in 20 °C for 28 days.

## 3.2 Test methods

### 3.2.1 High temperature test

The specimens were heated using an electric oven and the heating temperatures were set to ambient temperature, 100, 200 and 300 °C respectively. The temperature rising rate during the heating experiment was set to 5 °C /min and the temperature was kept constant for 5 hours after the oven temperature reached the target temperature in order to make sure the uniform heating throughout the whole specimens.

The top and bottom surfaces of the specimens were covered with ceramic fiber blanket which can tolerate 1000 °C high temperature as shown in Figure 3.4 during the heating test in order to protect the exposed rebar and to make sure that the heat was transferred through the side surface of the specimens.



Figure 3.4 Ceramic fiber blanket.

The residual compressive strength of the cylinder specimens was measured as shown in Figure 3.5 after the specimens were naturally cooled in air for 24 hours. In addition, the porosity of the high temperature damaged prism specimens was also measured in order to evaluate the deterioration of the concrete micro-structure.

$$P = \frac{W_s - W_d}{W_s - W_b} \times 100 \quad (3.1)$$

The porosity of the concrete specimens was calculated by using Equation 3.1. Where  $P$  is the porosity of the concrete specimen,  $W_s$  and  $W_b$  are the water submerged mass of the concrete specimen in air and in water,  $W_d$  is the oven dry mass of the concrete specimen in air.



Figure 3.5 Residual compressive strength test system.

### 3.2.2 Cyclic immersion into salt water and drying

The RC prism specimens were subjected to the cyclic immersion into salt water and drying in order to accelerate the chloride-induced steel corrosion process.

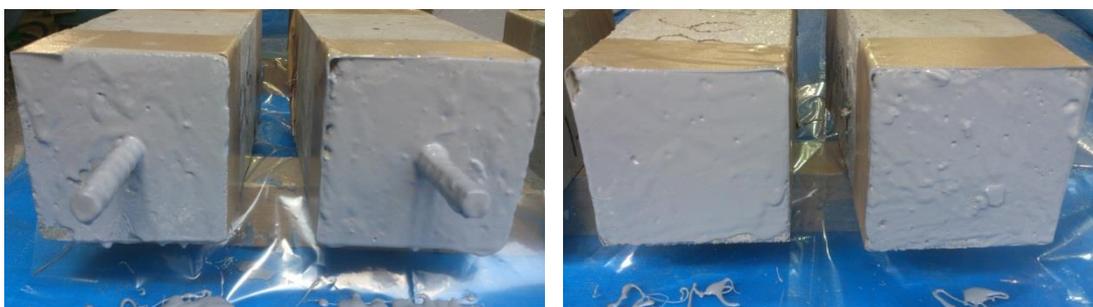


Figure 3.6 Epoxy resin protection on top and bottom surfaces.

The top and bottom surfaces of the prism specimens (which were protected using the ceramic fiber blanket during the heating test) were coated with epoxy resin as shown in Figure 3.6 before immersion into salt

water in order to make sure that the chloride ions (Cl<sup>-</sup>) will penetrate into the concrete through the side surface. The cycles of immersing the specimens in 10% NaCl solution for 4 days as shown in Figure 3.7 followed by drying the specimens in air for 3 days repeated for 90 days.



Figure 3.7 Specimens immersed in 10% NaCl solution.

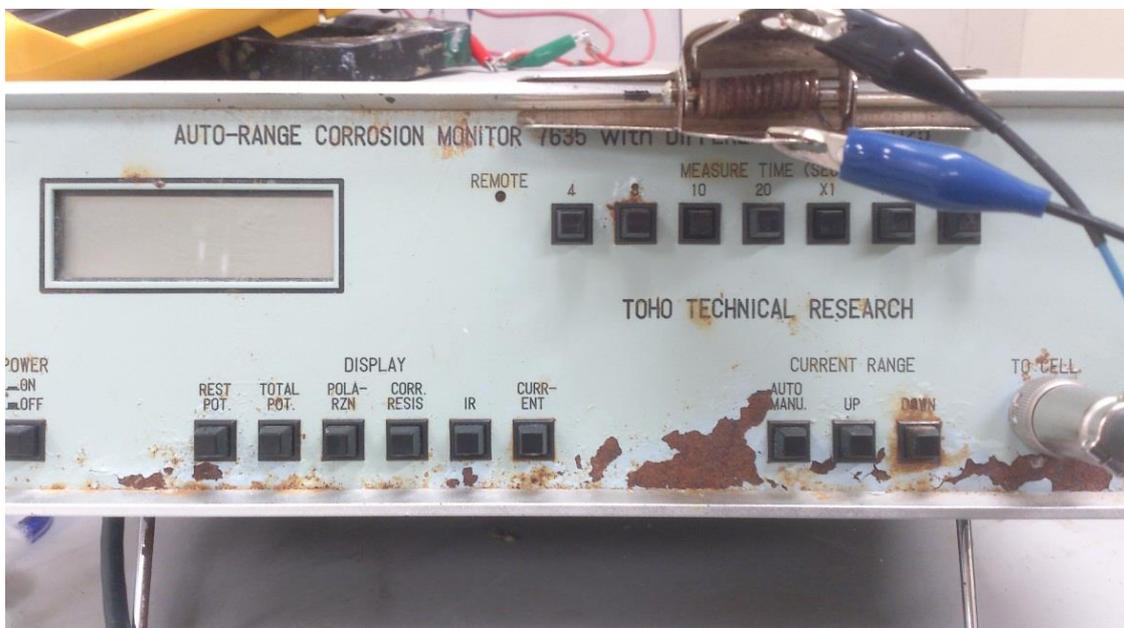


Figure 3.8 Electrochemical monitoring instrument.

The half-cell potential, polarization resistance and concrete resistance of the specimens were measured using the Auto-ranged Corrosion Monitor as shown in Figure 3.8 regularly during the test to monitor the rebar corrosion situation.

As a reference electrode for these electrochemical monitoring, saturated silver chloride (Ag/AgCl) was used. Polarization resistance was measured by the rectangular wave electric current polarization method, as the deference of impedances at 800 Hz and 0.1 Hz of electric current frequency. Resistivity was obtained as the impedance at 800 Hz of electric current frequency.

Table 3.2 List of specimens.

Name	Type of specimen	Heating temperature ( °C)	W/C (%)	Number
PA45	Prism	Ambient temperature	45	3
PA60	Prism	Ambient temperature	60	3
CA45	Cylinder	Ambient temperature	45	3
CA60	Cylinder	Ambient temperature	60	3
P145	Prism	100	45	3
P160	Prism	100	60	3
C145	Cylinder	100	45	3
C160	Cylinder	100	60	3
P245	Prism	200	45	3
P260	Prism	200	60	3
C245	Cylinder	200	45	3
C260	Cylinder	200	60	3
P345	Prism	300	45	3
P360	Prism	300	60	3
C345	Cylinder	300	45	3
C360	Cylinder	300	60	3

The distribution profiles of Cl<sup>-</sup> content in the concrete specimens were measured just after finishing the cyclic immersion into salt water and drying using the powder samples grinded from the each of five plates in a specimen as shown in Figure 3.9. The water soluble Cl<sup>-</sup> content and total Cl<sup>-</sup> content were measured by following to JIS A 1154. SEM (Scanning Electron Microscope) analysis observation of split concrete surface and the

measurement of weight loss of steel due to corrosion were also carried out after the cyclic immersion test. The weight loss of steel was measured by following to JCI-SC1. The list of the specimens is shown in Table 3.2.

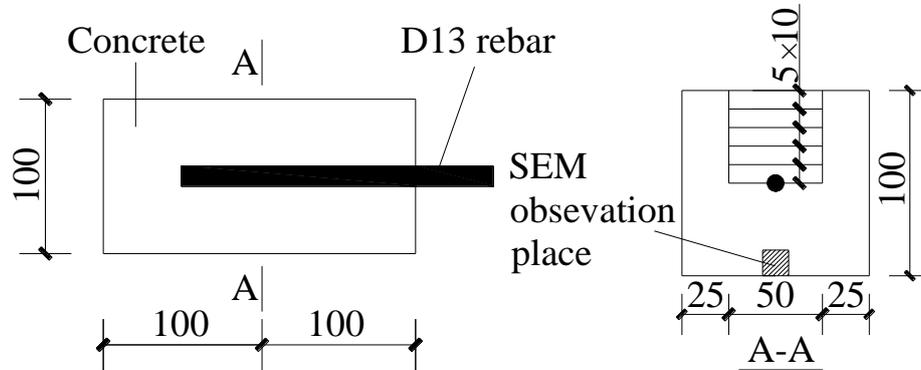
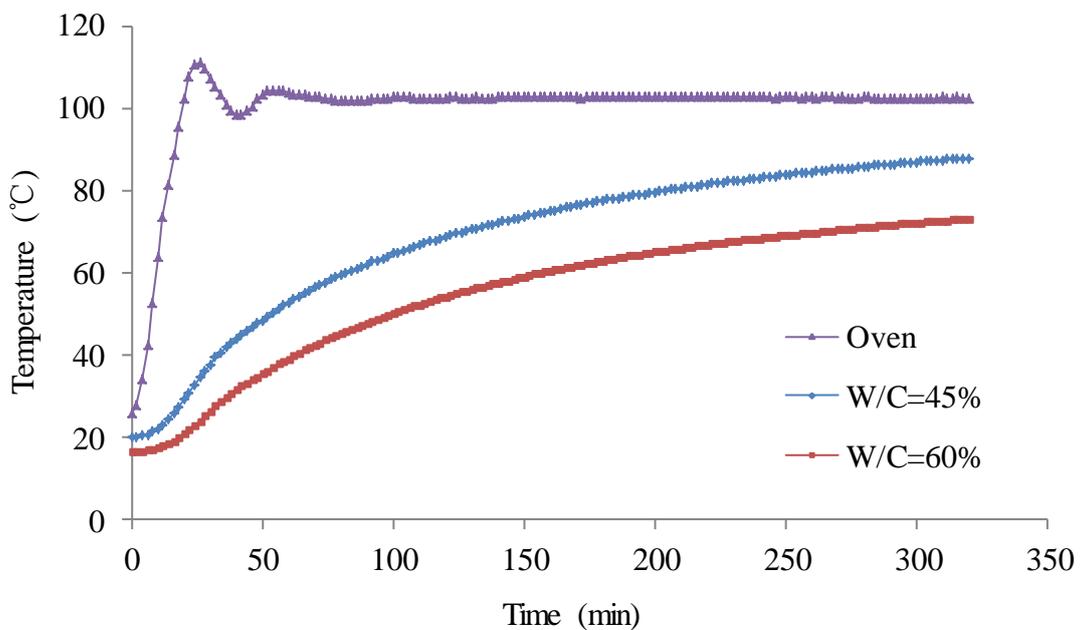


Figure 3.9 Cutting of the concrete for chemical analysis. (Unit: mm)

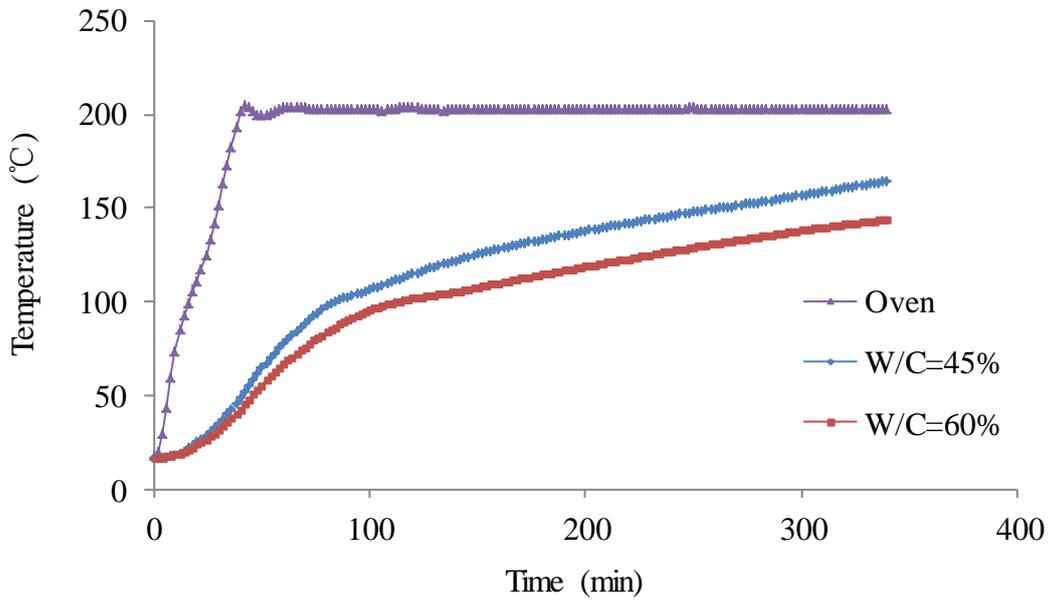
### 3.3 Results and discussion

#### 3.3.1 Variation curves of temperature in concrete during heating

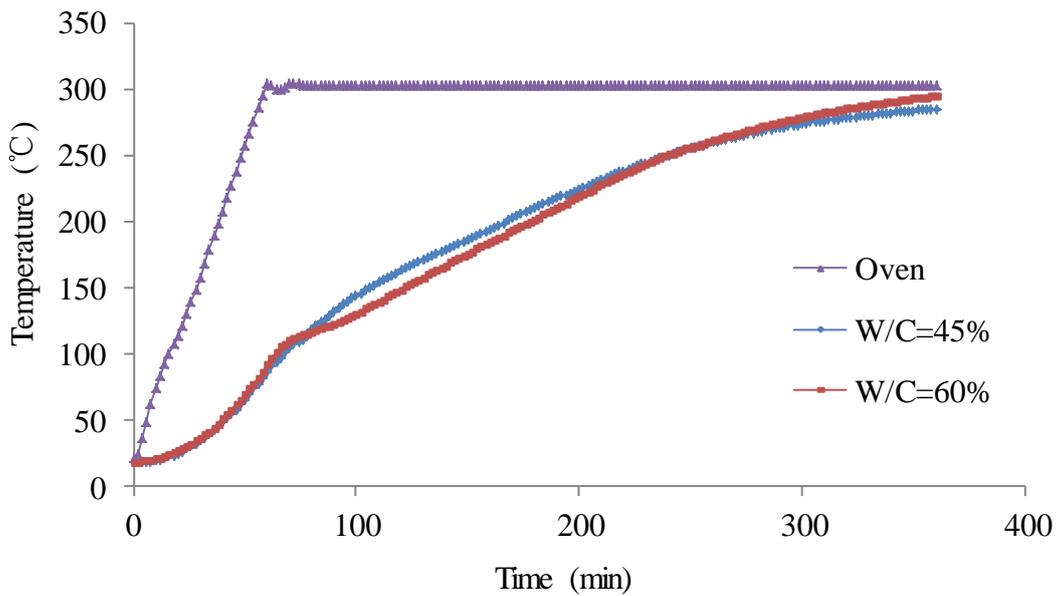
As shown in Figure 3.1, three thermocouples were embedded in concrete specimens at the different depth respectively. However, since the differences among the data measured by these three thermocouples were not remarkable, it can be said that the temperature was uniformly distributed in the concrete during the heating.



(a) Heating oven up to 100 °C



(b) Heating oven up to 200 °C



(c) Heating oven up to 300 °C

Figure 3.10 Variation curves of temperature in concrete.

Variation curves of temperature in concrete specimens during the heating are shown in Figure 3.10. It can be seen from the curves that the temperature of all kinds of concrete are rapidly increased from 20 °C to 100 °C. However, the rising rate of the temperature in concrete decreases after 100 °C and generates a temperature platform for all kinds of concrete. The

reason is been considered that the water in concrete will be evaporated when the temperature exceeds 100 °C, and the evaporation of water consumes some heat in concrete. In addition, as the heating temperature increased, the temperature of the concrete specimens after 4 hours' heating is also get closer to the target temperature.

On the other hand, in the cases of the 45% W/C concrete, the rising rate of temperature is higher than that of the cases of 60% W/C concrete for the 100 and 200 °C heated specimens, whereas this phenomenon is not found for the 300 °C heated specimens. The reason is that since the amount of water in 60 % concrete is more than the case of 45 % concrete, the rising rate of temperature in 60 % concrete becomes relatively low due to the water evaporation effect when the surrounding temperature is 100 or 200 °C, whereas in the cases of 300 °C heated specimens, the effect of water evaporation becomes minor due to the relatively early convergence of the water evaporation.

### 3.3.2 Residual compressive strength of concrete after heating

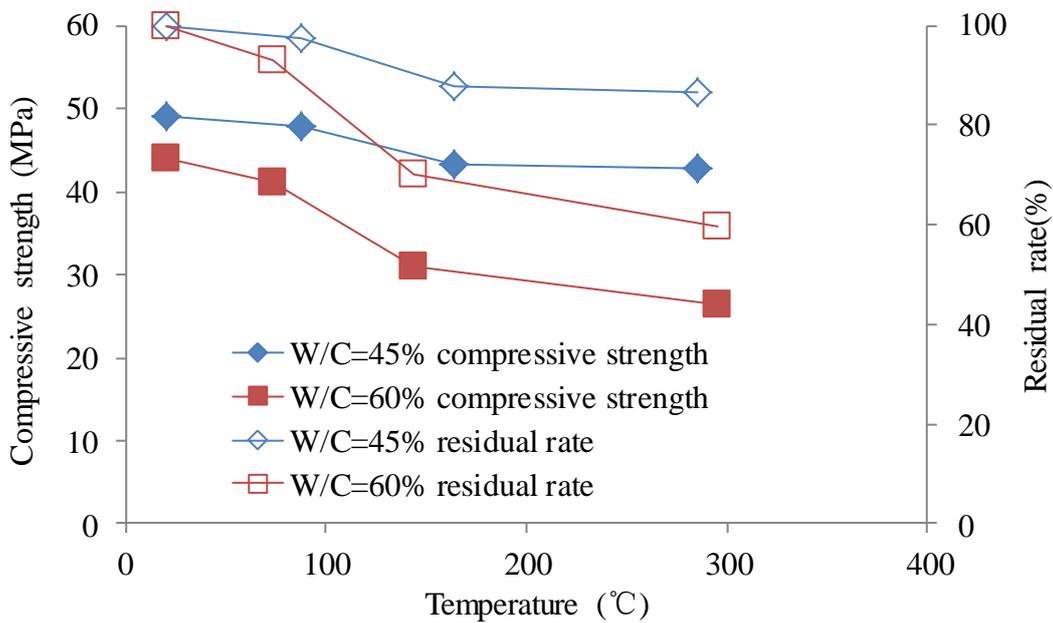


Figure 3.11 Residual compressive strength of concrete after heating.

The residual compressive strength of the concrete specimens after the heating test was measure after the specimens were cooled naturally in air for 24 hours. The residual compressive strength and the residual rate of the concrete specimens after the heating are shown in Figure 3.11. It can be seen from Figure 3.11 that the residual compressive strength of the concrete

decreases when the heating temperature exceeds 100 °C due to the developing of micro-cracks in concrete caused by the high vapor pressure and the thermal stress. The vapor stress was caused by the evaporation of water in concrete and the thermal stress was caused by the different thermal expansions between the aggregates and cement paste.

However, the additional decrease of the residual compressive strength of the high temperature damaged concrete when the heating temperature reaches around 300 °C is relatively small. The similar results have been reported in the previous researches with the possible mechanism of the re-hydration of the cement paste due to the steam curing in concrete subjected to the heating to around 300 °C [1-4]. This re-hydration of the cement paste in the powder rich concrete such as self-compacting concrete, high-strength concrete and high-performance concrete will cause the increase of the residual compressive strength when the heating temperature is not too high.

The decrease of the residual compressive strength of 60% W/C concrete is larger than that of 45% W/C concrete. As shown in Table 1, as the cement content of the 45 % W/C concrete is larger than that of the 60 % W/C concrete, the steam curing effect during the heating in the 45 % W/C concrete could be more effective than the case of the 60 % W/C concrete, while the damage of the 60 % W/C concrete due to the micro-cracks formed by the high vapor pressure of the moisture in concrete and the thermal stress would be remained after the heating and affect the mechanical properties significantly.

In addition, the cylinder specimens after the compressive strength test were also sprayed with phenolphthalein solution to investigate the carbonation situation of the high temperature damaged concrete but the carbonation of concrete was not observed in all specimens. From this result, it can be said that the component of  $\text{Ca}(\text{OH})_2$  in the concrete didn't be decomposed by the heating conducted in this study.

### 3.3.3 Porosity of concrete after heating

The porosity of concrete after heating was measured according to Equation 3.1 and the porosity variations of the concrete are shown in Figure 3.12. It can be seen from Figure 3.12 that the porosity significantly increases when the heating temperature exceeds 100 °C due to the development of micro-cracks in concrete caused by the high vapor stress

and different thermal expansion between aggregates and cement paste.

However, the additional increase of the porosity observed around 200 °C to 300 °C is not significant compared with the case of before 200 °C which is caused by the re-hydration of the cement paste in concrete. Such a tendency of the porosity increase with the rise in temperature is also agree with the tendency of the decrease of the residual compressive strength as shown in Figure 3.11.

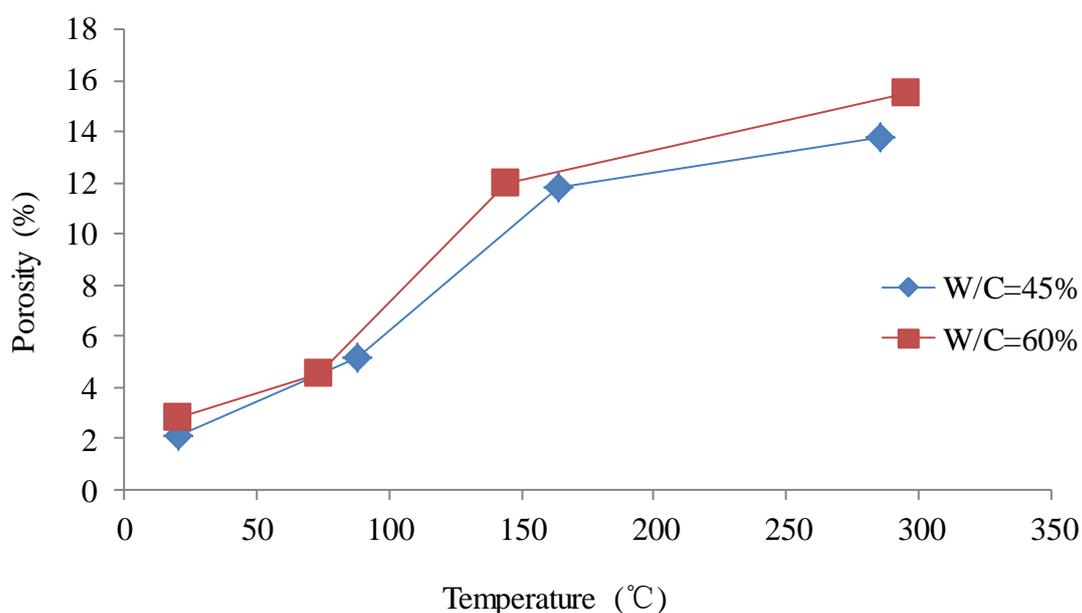
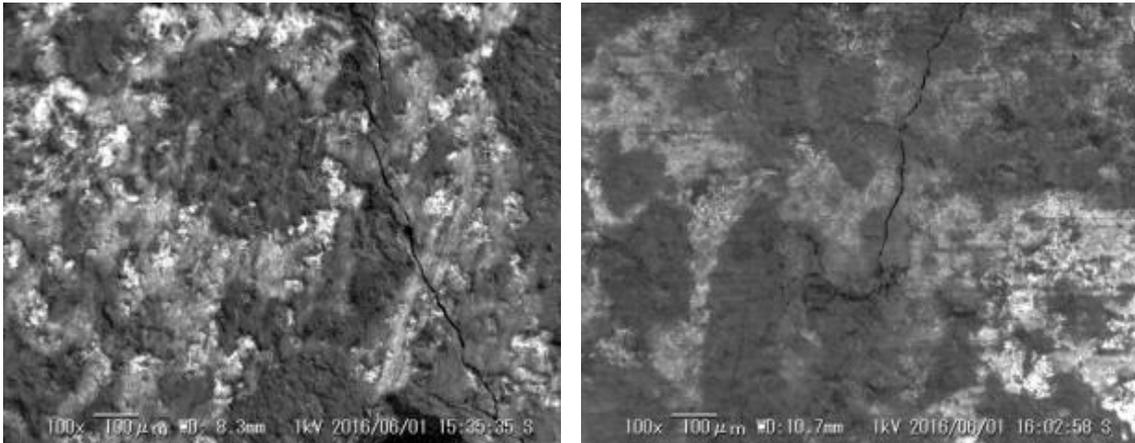


Figure 3.12 Porosity of concrete after heating.

### 3.3.4 SEM observation of concrete

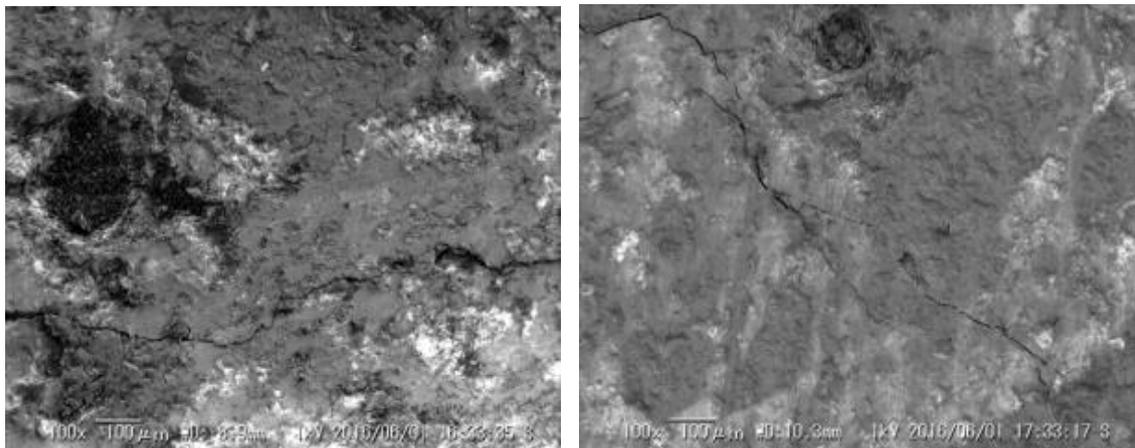
Results of SEM observation of the concrete (the sampling point in a specimen is shown in Figure 3.9) after the heating and the cyclic immersion into salt water and drying are shown in Figure 3.13. The observation magnification of the SEM was set to 100x and it can be seen from the figures that compared with the concrete section of the un-heated specimens, concrete section heated to over 100 °C ((a) and (b) in Figure 3.13) became rough and small cracks were also observed, and the concrete section heated around 300 °C ((c) and (d) in Figure 3.13) became porous and larger cracks were observed. The results of the SEM observation indicate that the micro-structure of concrete changed to porous and micro-cracks were introduced even if the affected temperature was around 150 °C. These micro-cracks were generated by the high vapor stress and the different

thermal expansion between the aggregates and the cement paste. Such damage of concrete micro-structure would cause the decrease of the residual compressive strength as shown in 3.3.2 and also could affect the resistance performance of concrete against the penetration of ions and steel corrosion behavior described in the following parts.



(a) P245

(b) P260



(c) P345

(d) P360

Figure 3.13 SEM photos of micro-structure of the concrete after heating and cyclic immersion into salt water and drying.

### 3.3.5 Distribution of $\text{Cl}^-$ in concrete

Distribution of water soluble  $\text{Cl}^-$  content, total  $\text{Cl}^-$  content and the ratio of water soluble  $\text{Cl}^-$  to total  $\text{Cl}^-$  content in concrete after the heating test and the cyclic immersion into salt water and drying are shown in Figure 3.14 to 3.16 respectively.

According to Figure 3.14 and Figure 3.15, the specimens heated to

around 200 °C (P245 and P260) or 300 °C (P345 and P360) show the relatively higher Cl<sup>-</sup> content compared with the cases of un-heated specimens (PA45 and PA60) or specimens heated to lower than 100 °C (P145 and P160). Especially in the cases of specimens of 60% W/C heated to around 200 °C or 300 °C, the declination of Cl<sup>-</sup> content with the depth around the steel bar becomes mild and then the Cl<sup>-</sup> content around the steel bar becomes relatively high. The high Cl<sup>-</sup> content indicates the severe development of micro-cracks in concrete caused by the heating and the results were consistent with the porosity as shown in 3.3.3.

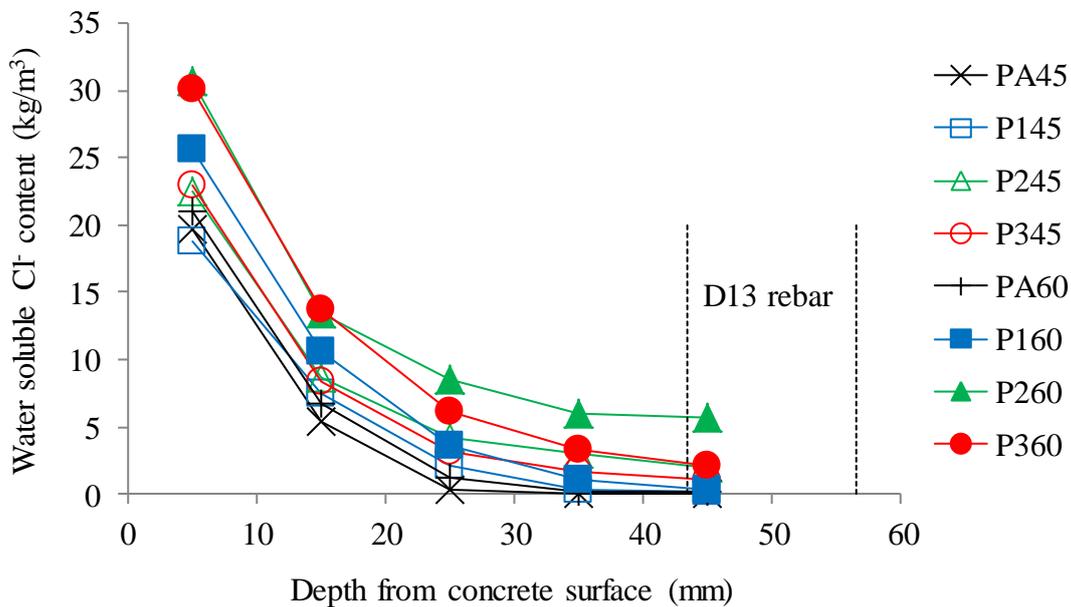


Figure 3.14 Distributions of water soluble Cl<sup>-</sup> content.

Moreover, Cl<sup>-</sup> content values in 200 °C heated specimens are higher than those in the 300 °C heated specimens for both 45% W/C and 60% W/C concrete. The reason of this is considered that the influence of steam curing effect on the resistance performance of concrete against Cl<sup>-</sup> penetration is larger for the 300 °C heated concrete specimens than that of the 200 °C heated concrete specimens.

In addition, regarding W/C of the concrete, the Cl<sup>-</sup> content values in the 60% W/C concrete were higher than those in the 45% W/C concrete regardless of the heating temperature. The reason is considered that the 60% W/C concrete contains more water than that of the 45% W/C concrete and the higher water content will generate higher vapor pressure under high temperature, which will generate more micro-cracks and make the Cl<sup>-</sup>

penetrate into concrete more easily.

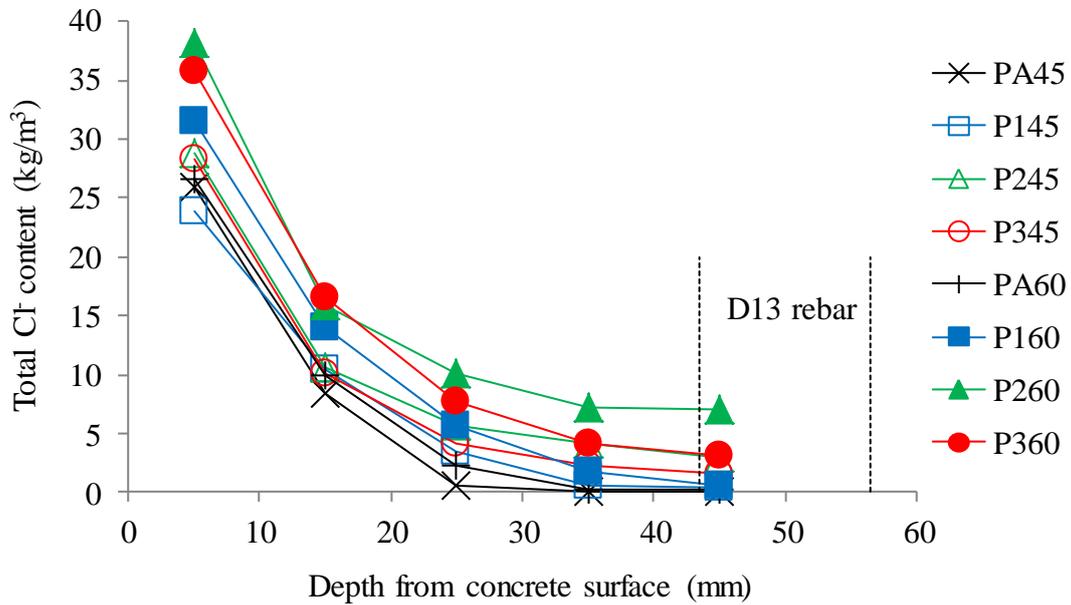


Figure 3.15 Distributions of total Cl<sup>-</sup> content

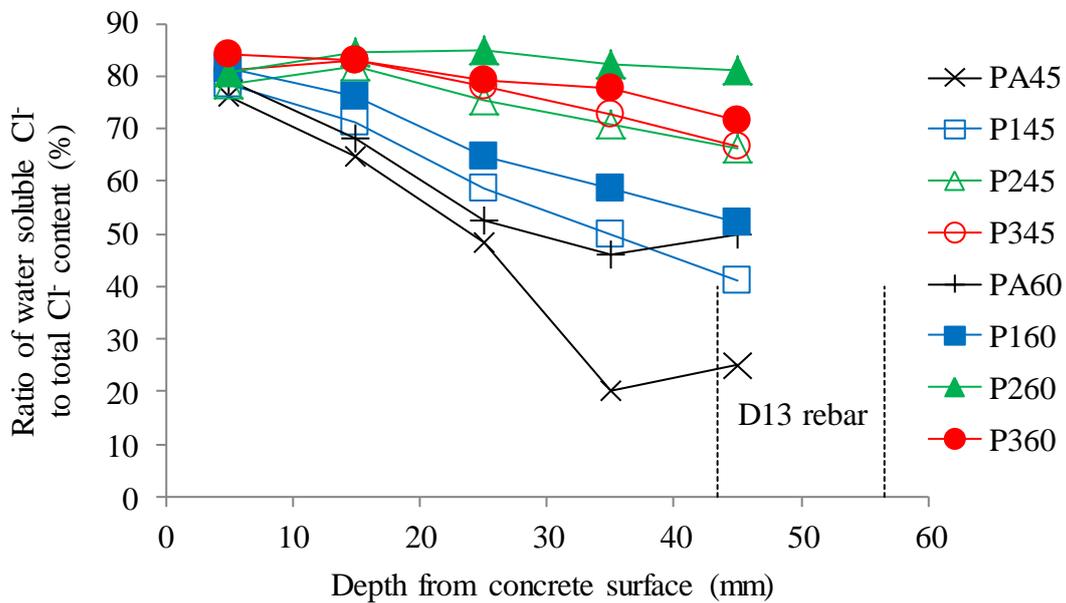


Figure 3.16 Distributions of ratio of water soluble Cl<sup>-</sup> to total Cl<sup>-</sup>.

Generally, Cl<sup>-</sup> ions penetrate from the concrete surface as the apparent diffusion process with the fixing of a part of free Cl<sup>-</sup> to the cement hydration products. However, in the cases of the concrete heated to around 200 °C or 300 °C, the penetration of Cl<sup>-</sup> seems different from the usual

diffusion process observed in the general cases. Similar  $\text{Cl}^-$  profiles in concrete subjected to the heating to around 300 °C were also reported in the previous researches [5-7] and such  $\text{Cl}^-$  profiles will promote the steel corrosion in concrete. Figure 3.16 shows the variations of the ratio of water soluble to total  $\text{Cl}^-$  content. It can be seen from Figure 3.16 that the ratio of water soluble to total  $\text{Cl}^-$  content increases with the rise in the heating temperature, which means that the concrete ability of fixing  $\text{Cl}^-$  in the hydration products like Friedel's salt was significantly decreased by the heating of the concrete.

From these results, it can be said that the promotion of  $\text{Cl}^-$  penetration into concrete affected by the heating to around 200 or 300 °C shown in Figure 3.14 and Figure 3.15 would be caused not only by the damage of the micro-structure in the heated concrete but also by the decrease of the  $\text{Cl}^-$  fixing ability of the concrete due to the heating. Such phenomena would accelerate not only  $\text{Cl}^-$  penetration into concrete but also steel corrosion in concrete after the heating.

On the other hand, the ratio of the water soluble to total  $\text{Cl}^-$  content of 60% W/C concrete is also higher than that of the cases of the 45% W/C concrete despite the heating temperature, which indicates that the higher water content in concrete will lead to higher water soluble  $\text{Cl}^-$  content and will eventually cause the severer steel corrosion situation in the high temperature damaged concrete.

### 3.3.6 Apparent diffusion coefficient of $\text{Cl}^-$ in concrete

Variation curves of apparent diffusion coefficients of  $\text{Cl}^-$  in the high temperature damaged concrete calculated by using Equation 3.2 with the heating temperature are shown in Figure 3.17.

$$C(x,t) - C_i = C_s \left[ 1 - \text{erf} \left( \frac{x}{2\sqrt{Dt}} \right) \right] \quad (3.2)$$

Where  $C(x,t)$  is the  $\text{Cl}^-$  concentration at distance  $x$  from the exposed concrete surface after time  $t$ ,  $C_i$  is the original  $\text{Cl}^-$  concentration,  $C_s$  is the  $\text{Cl}^-$  concentration of the exposed concrete surface,  $x$  is the distance from the exposed concrete surface,  $t$  is the exposure time,  $D$  is the  $\text{Cl}^-$  diffusion coefficient and  $\text{erf}$  is the error function.

It can be seen from Figure 3.17 that in the cases of 60 % W/C concrete, the diffusion coefficient values increase with the rise in the heating

temperature, while in the cases of 45 % W/C concrete, the diffusion coefficient increases with the heating to around 100 °C but it decreases with the heating to around 200 °C. Since the 45 % W/C concrete contains much amount of un-hydrated cement compared with the case of 60 % W/C concrete, the steam curing effect would be effective to reduce the diffusion coefficient of the heated concrete as described in 3.3.2. However, as shown in 3.3.5, considering the change in the Cl<sup>-</sup> fixing ability of the concrete due to the heating, it may be difficult to explain the Cl<sup>-</sup> penetration property of concrete after heating by using the diffusion process.

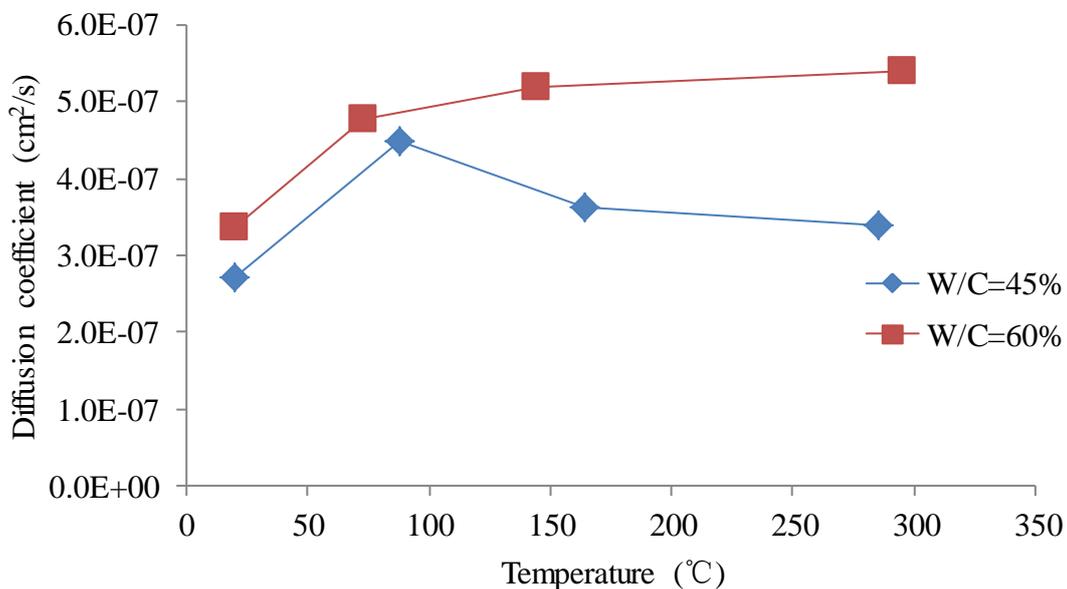


Figure 3.17 Variation curves of apparent diffusion coefficients of Cl<sup>-</sup> with the heating temperature.

### 3.3.7 Variation of electrochemical indexes

Variation curves of half-cell potential, polarization resistance and concrete resistance of RC specimens during the cyclic immersion into salt water and drying are shown in Figure 3.18 to 3.20 respectively.

Referring to the criteria of steel corrosion in concrete shown in ASTM C876-91, the potential nobler than -0.09 V to saturated Ag/AgCl as the reference electrode indicates non-corrosion with a probability of 90% or more, the potential lower than -0.24 V indicates corrosion with a probability of 90% or more, and the potential between them means uncertain about the corrosion state. These criteria are also shown in Figure 3.18.

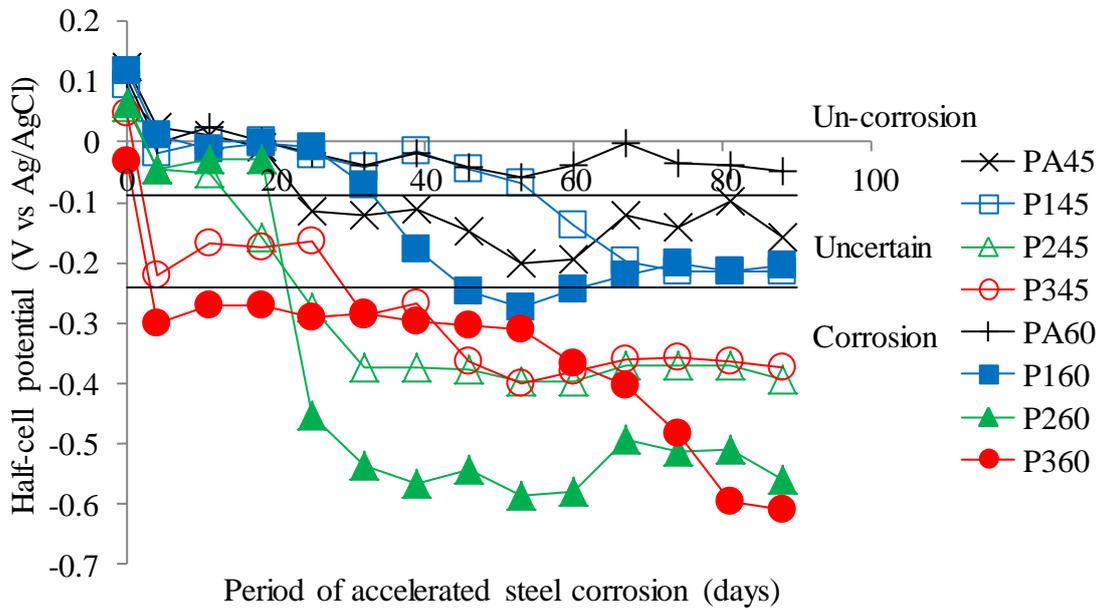


Figure 3.18 Variation curves of half-cell potentials during cyclic immersion into salt water and drying.

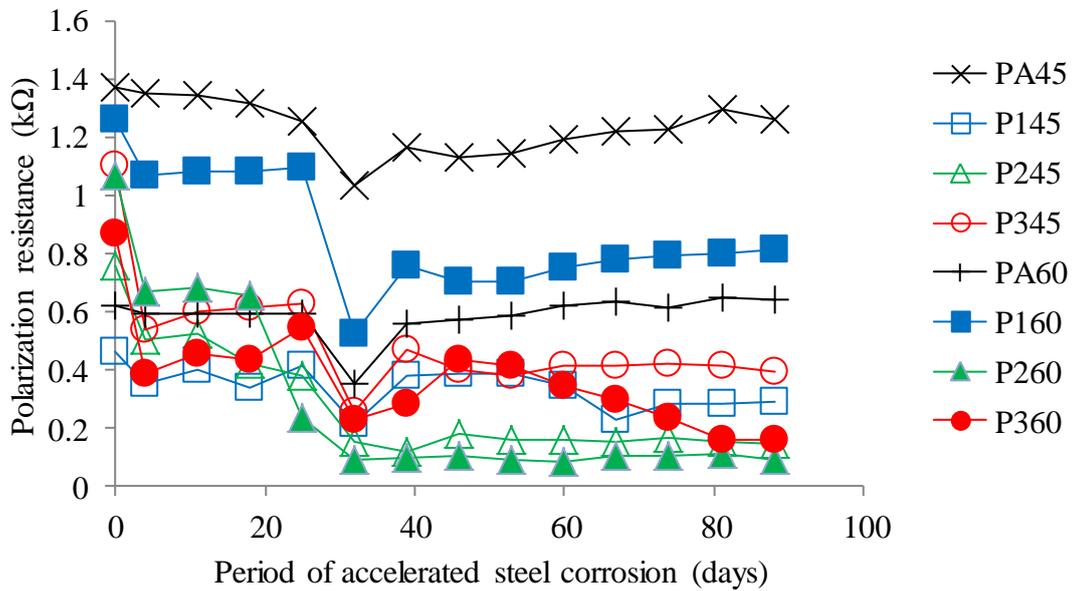


Figure 3.19 Variation curves of polarization resistance during cyclic immersion into salt water and drying.

From Figure 3.18, it can be seen that the half-cell potential values of the non-heated and 100 °C heated concrete specimens decrease slowly during the period of the accelerated steel corrosion and almost keep the range of

the uncertain area. On the other hand, the half-cell potential values of the 200 or 300 °C heated specimens rapidly decrease during the cyclic immersion into salt water and drying due to the destruction of the micro-structure caused by the high temperature. These cases are classified to the corrosion area in the ASTM criteria but the half-cell potential values of 45 % W/C cases are relatively nobler than the cases of 60 % W/C, which indicates that higher cement content can improve the Cl<sup>-</sup> resistance performance due to the re-hydration if the heating temperature is not exceed 300 °C. These results are also consistent with the results of the porosity or the Cl<sup>-</sup> content profiles in the concrete as discussed in 3.3.3 and 3.3.5.

The polarization resistance  $R_p$  is known as an electrochemical index that can be used to calculate the steel corrosion rate in concrete as shown in Equation 3.3.

$$I_{corr} = k/(R_p) \quad (3.3)$$

The equation expresses that the inverse of  $R_p$  is proportional to the corrosion current density  $I_{corr}$  as the steel corrosion rate, so a quantitative estimation of corrosion progress in concrete is possible with using an appropriate constant  $k$ .

It can be seen from Figure 3.19 that the polarization resistance values of the specimens heated to around 200 or 300 °C are all decreasing during the period of the accelerated steel corrosion. However, the polarization resistance values of the un-heated specimens maintained almost constant during the accelerated steel corrosion test. It can be obtained from this experimental result that the steel corrosion rates of the specimens heated to around 200 or 300 °C increase with the accelerated steel corrosion whereas the steel corrosion rates of the un-heated concrete specimens keep constant during the accelerated corrosion test.

On the other hand, the decrease of the polarization resistance of the concrete specimens heated to around 200 °C is larger than that of the concrete specimens heated to around 300 °C. The reason is considered that the re-hydration process of the cement paste occurred in the 300 °C heated specimens is heavier than that of the 200 °C specimens due to the steam curing effect. Such a tendency is also consistent with the results of Cl<sup>-</sup> distribution in concrete as shown in Figure 3.14 and Figure 3.15.

The concrete resistivity is an electrical resistance of the cover concrete and also an index depending on the moisture or the Cl<sup>-</sup> content in concrete, which affects the conductivity of the steel corrosion current.

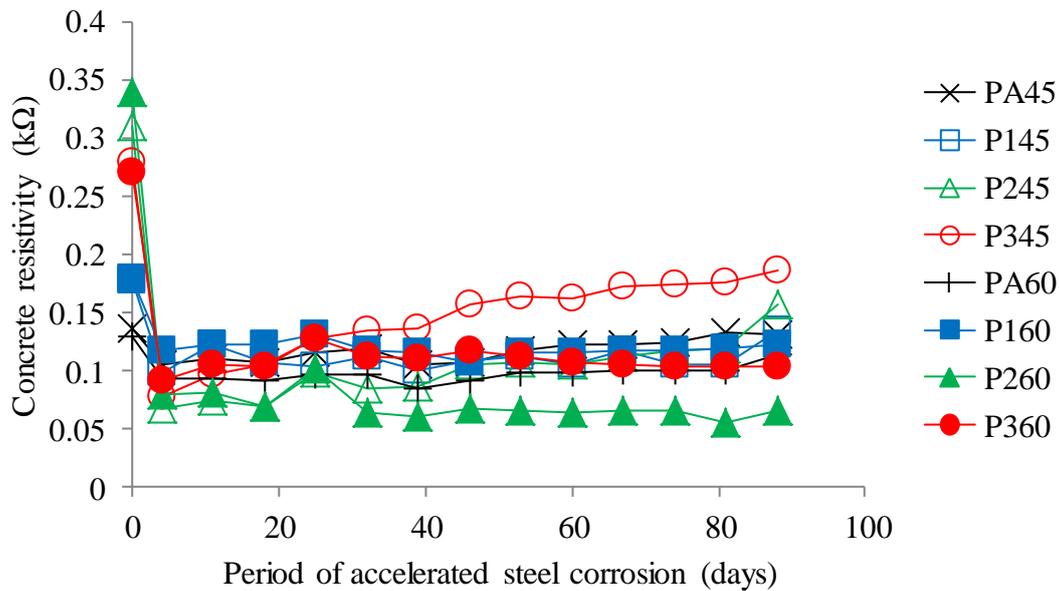


Figure 3.20 Variation curves of concrete resistivity of the prism specimens during cyclic immersion into salt water and drying.

According to Figure 3.20, it can be seen that the concrete resistivity values measured before starting the cyclic immersion into salt water and drying increase with the heating temperature. The reason is considered to relate to the drying state of the concrete specimens, which means that the higher heating temperature will lead to the drier situation of the concrete and eventually cause the higher concrete resistivity. After starting the immersion into salt water, the concrete resistivity values decrease with the penetration of salt water for all kinds of concrete.

On the other hand, the 200 °C heated 60% W/C specimen (P260) shows gradual decrease of the concrete resistivity while 300 °C heated 45% W/C specimen (P345) shows gradual increase of the concrete resistivity during the period of the accelerated steel corrosion test. These results suggest that P260 specimens got the heaviest damage of the micro-structure in concrete due to the heating and P345 got the largest effect of the steam curing, which are confirmed by the results of porosity, chloride penetration tendency and other electrochemical indexes.

### 3.3.8 Weight loss of steel in concrete due to corrosion

The steel bars used in this program were removed from the concrete specimens after the accelerated steel bar corrosion experiment and the weight of the steel bars were also measured. Then the steel bars were immersed in 10% Diammonium Hydrogen Citrate (Ammonium Citrate

Dibasic) solution and kept constant at 60 °C for 24 hours in order to remove the corrosion rust. After that, the steel bars were washed using water and the weight of the washed steel bars was measured again to calculate the weight losses of the steel bars during the corrosion experiment.



Figure 3.21 Steel bars after corrosion test.

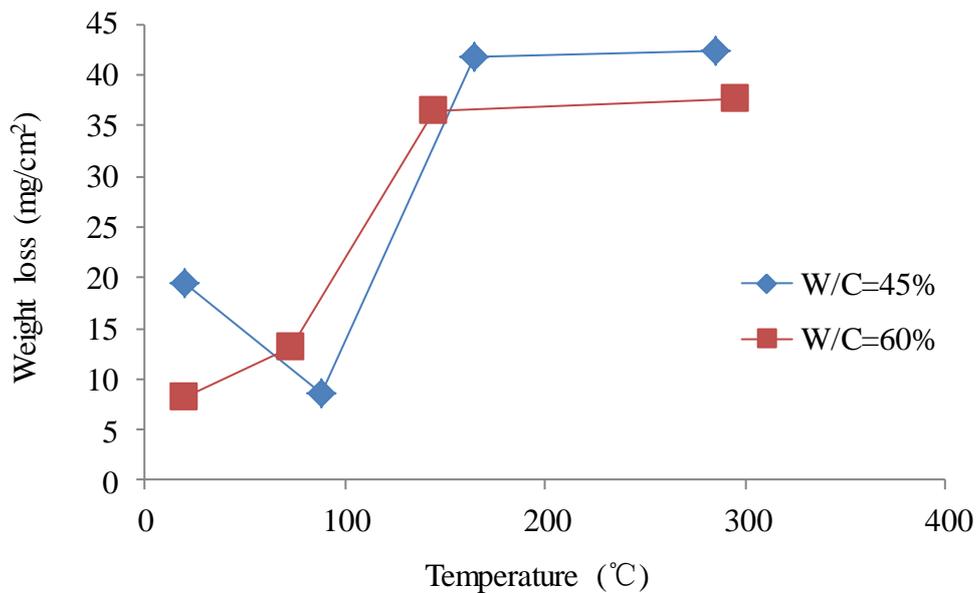


Figure 3.22 Weight loss of steel due to corrosion.

The situation of the steel bars after the accelerated corrosion experiment are shown in Figure 3.21 and the weight loss data of steel bars removed from the specimens after the cyclic immersion into salt water and drying are shown in Figure 3.22 respectively.

From Figure 3.22, it can be seen that the weight loss values of the steel bars in concrete significantly increase when the heating temperature exceeds 100 °C and maintain the severe corrosion level from around 200 °C to 300 °C regardless of the difference of the concrete W/C, these results are also consistent with the results of the Cl<sup>-</sup> distribution and electrochemical indexes as shown in 3.3.5 and 3.3.7. Namely, when the heating temperature exceeds 100 °C and rise to 200 or 300 °C, the damage of the concrete micro-structure and the decrease of the Cl<sup>-</sup> fixing ability of the concrete accelerated the Cl<sup>-</sup> penetration after the heating, which results in the acceleration of the steel corrosion in concrete.

Considering the results of this study, even if the decrease of the compressive strength with the heating is not so significant if the heating temperature is not too high, the corrosion resistance performance of concrete after the heating could be severely decreased. So, the serviceability of the fire-damaged concrete structure should be judged including the reduction of the durability after the heating [8-10].

### 3.4 Summary

The experimental results obtained from this chapter can be summarized as follows:

(1) The compressive strength of the concrete decreased when the heating temperature exceeded 100 °C whereas the additional decrease of the compressive strength when the temperature reached around 300 °C was relatively small. In addition, the decrease of compressive strength of 60% W/C concrete was larger than that of 45% W/C concrete.

(2) The porosity increased when the heating temperature exceeded 100 °C whereas the additional increase of porosity observed around 200 °C to 300 °C was not significant. Such a tendency of the porosity increase with the rise in temperature is also agree with the tendency of the decrease of the compressive strength.

(3) The Cl<sup>-</sup> penetration into concrete after the heating to around 200 or 300 °C was accelerated not only by the damage of the micro-structure in the heated concrete but also by the decrease of the Cl<sup>-</sup> fixing ability of the concrete due to the heating.

(4) The steel corrosion rates of the specimens heated to around 200 or 300 °C increased with the accelerated steel corrosion compared with the cases of the un-heated specimens. On the other hand, the decrease of the polarization resistance of the specimens heated to around 200 °C was larger than that of the specimens heated to around 300 °C. Such a tendency is also observed in the results of the concrete resistivity especially in the cases of the 45 % W/C concrete.

(5) When the heating temperature exceeded 100 °C and rose to 200 or 300 °C, the weight loss of the steel in concrete due to the chloride-induced corrosion was accelerated.

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## Chapter 4 Experimental research on the properties of self-compacting concrete containing copper slag aggregate after heating up to 400 °C

In this chapter, the residual mechanical properties and the durability against chloride attack of the self-compacting concrete containing copper slag fine aggregate after heating up to 400 °C were investigated. Cylinder concrete specimens were prepared and heated to 100, 200, 300 and 400 °C respectively, un-heated specimens were also prepared in order to make a comparison. After that, residual compressive strength, splitting tensile strength, elastic modulus and stress-strain curve of the concrete specimens were measured with the change in the porosity of concrete. Moreover, the resistance performance against the penetration of  $\text{Cl}^-$  (chloride ions) into the concrete after the heating was also evaluated. In addition, the micro-structure around the fine aggregate in the specimens was also observed by using SEM (scanning electron microscope).

### 4.1 Materials and specimens

#### 4.1.1 Mixture proportions

Mixture proportions of the self-compacting concrete used in this program are shown in Table 4.1. All the mixtures were designed to have a constant binder content of  $500 \text{ kg/m}^3$ . Water to binder ratio (W/B) was set to 40%. Ordinary Portland cement (density:  $3.16 \text{ g/cm}^3$ , R2O: 0.56%), JIS A6021 Type-II fly ash (density:  $1.95 \text{ g/cm}^3$ , surface area:  $2500 \text{ cm}^2/\text{g}$ ), normal fine aggregate (density:  $2.57 \text{ g/cm}^3$ , water absorption: 1.77%) and normal coarse aggregate (density:  $2.57 \text{ g/cm}^3$ , water absorption: 1.55%, maximum size: 15 mm) were used. Superplasticizer (MasterGlenium SP8SV X2) was also used in this program in order to improve the fluidity performance of the concrete.

Copper slag (CUS) is a by-product generated during the process of copper extraction by smelting. During smelting, impurities become slag which floats on the molten metal. Slag that is quenched in water produces angular granules which are disposed of as waste or utilized. Production of one ton of copper produces about three tons of copper slag and about 24.6 million tons of copper slag is generated from world copper production every year and the annual amount of copper slag in Japan is about two million tons. Copper slag is mainly used for surface blast-cleaning. Abrasive blasting is used to clean and shape the surface of metal, stone, concrete and other materials. The blasting media manufactured from copper

slag brings less harm to people and environment than sand. The product meets the most rigid health and ecological standards. The recycled copper slag can be used in concrete as a substitute for fine aggregate and improve the fluidity of the concrete, while the mixing of the copper slag aggregate also would promote the bleeding of the fresh concrete due to the high density and glassy smooth surface of the copper slag. For that reason, mixing copper slag aggregate into powder-rich concrete such as self-compacting concrete and high performance concrete might be effectively to reduce the bleeding of the fresh concrete.

The copper slag aggregate used in this study was produced at Onahama in Japan (density: 3.50 g/cm<sup>3</sup>, water absorption: 0.1%). The maximum size of the copper slag aggregate was 2.5 mm. The copper slag was used to replace fine aggregate in self-compacting concrete and the replacing ratios were set to 0, 20 and 40% respectively referring to the recommendation ratio by JSCE [1].

Table 4.1 Mixture proportions of concrete.

Name	Unit mass (kg/m <sup>3</sup> )							W/B (%)	Air (%)	SF (mm)
	W	C	FA	G	S	CUS	SP			
NC	200	300	200	860	845	0	9.0	40	1.4	665
CUS20	200	300	200	860	676	169	5.0	40	1.4	565
CUS40	200	300	200	860	507	338	4.0	40	1.7	615

FA-Fly ash, CUS-Copper slag, W/B-Water to binder ratio, SP-Superplasticizer, SF-Slump flow.

#### 4.1.2 Preparation of specimens

The slump flow and air content of fresh concrete are also shown in Table 4.1. All kinds of concrete showed the slump flow over 550 mm while a remarkable segregation was not observed. In the cases of mixing copper slag fine aggregate, the amount of SP agent (superplasticizer) was reduced from the case of normal aggregate concrete to the 40% CUS replaced concrete because of the fluidity promoting effect of the copper slag aggregate. As for the CUS20 and CUS40 concrete, although the SP agent was reduced from 5.0 kg/m<sup>3</sup> to 4.0 kg/m<sup>3</sup>, the slump flow of the fresh concrete was increased from 565 mm to 615 mm. From this point, we can say that the using of copper slag fine aggregate indeed improved the fluidity performance of the self-compacting concrete.

Cylinder specimens which size is  $\Phi 100 \times 200$  mm were casted and then the specimens were demolded 24 hours after the casting. All specimens were cured in water controlled constantly at 20 °C for 28 days and dried in air at 20 °C for 7 days before the heating experiment.

## 4.2 Test methods

### 4.2.1 Heating of specimens

According to the previous related papers, the residual compressive strength of concrete was severely deteriorated when the concrete is subjected to a high temperature environment over 500 °C [2-3]. It was also reported that the self-compacting concrete occurred severely explosive spalling when the heating temperature exceeded 400 °C due to its high density [4].

In order to investigate the residual properties for the strengthening and rehabilitation of the fire damaged self-compacting concrete constructions and to prevent the explosive spalling of concrete during the heating experiment, the heating temperatures were set to ambient temperature, 100, 200, 300 and 400 °C respectively in this program. The specimens were heated by using an electric oven and the temperature rising rate was set to 5 °C /min. The temperature was kept constant for 4 hours after the oven temperature reached the target temperature in order to make sure the uniform heating throughout the whole concrete specimens. All the concrete specimens were naturally cooled in air for 24 hours after the heating experiment.

### 4.2.2 Measurement of mass loss and porosity of concrete

Using the heated specimens, the mass loss (weight loss of the specimens due to the heating) and the porosity of concrete were measured after the specimens were cooled in air naturally for 24 hours.

The mass loss of the concrete specimens was calculated by measuring the weight of the specimens before and after the heating experiment.

The porosity of the concrete was calculated according to the following Equation 4.1:

$$P = \frac{W_s - W_d}{W_s - W_b} \times 100 \quad (4.1)$$

The porosity of the concrete specimens was calculated by using Equation 3.1. Where  $P$  is the porosity of the concrete specimen,  $W_s$  and  $W_b$  are the

water submerged mass of the concrete specimen in air and in water,  $W_d$  is the oven dry mass of the concrete specimen in air.

In addition, the micro-structure of the cement paste around the fine aggregate in concrete was also observed by using SEM after the compressive strength experiment. The developing pattern of the micro-cracks and voids around the copper slag aggregate and normal sand in concrete can help us to understand the deterioration process of the high temperature damaged concrete. The SEM observation samples used in this program were cut from the center part of the concrete specimens.

#### 4.2.3 Compressive strength test

Three specimens for each heating condition were subjected to the compressive strength test after the heating experiment and the residual compressive strength, elastic modulus and stress-strain curves of the specimens were measured by following JIS A 1008: 2006.

#### 4.2.4 $Cl^-$ penetration test

The top and bottom circular surfaces of the cylinder concrete specimen for  $Cl^-$  penetration test were coated with epoxy resin after the heating experiment in order to make sure that the  $Cl^-$  could only penetrate from the side surface into the specimens. Then the concrete specimens were immersed in 10% NaCl solution for one month.

After that, the concrete specimens were split after the immersion by following JIS A 1113: 2006 and the splitting tensile strength was also measured at that time.

At last, the  $Cl^-$  penetration depth was measured by using the method of spraying 0.1 mol/L  $AgNO_3$  solution referring to the method shown in the previous related paper [5].

### 4.3 Results and discussion

#### 4.3.1 Damage state and mass loss due to heating

Almost all specimens showed no damage in appearance after the heating up to 400 °C excepting one NC specimen exploded by the heating to 400 °C, which means that the mixing of the copper slag fine aggregate into the self-compacting concrete could reduce the susceptibility of explosive spalling of the self-compacting concrete. The reason of this is considered that the bleeding water gathered along the interface between the copper slag fine aggregate and the cement paste generated the micro-cracks and voids during the curing process. These micro-cracks and voids in the

self-compacting concrete would release the high vapor pressure caused by the evaporation of water during the heating. Hereinafter the data of the concrete specimen damaged by the explosive spalling are not included in the results.

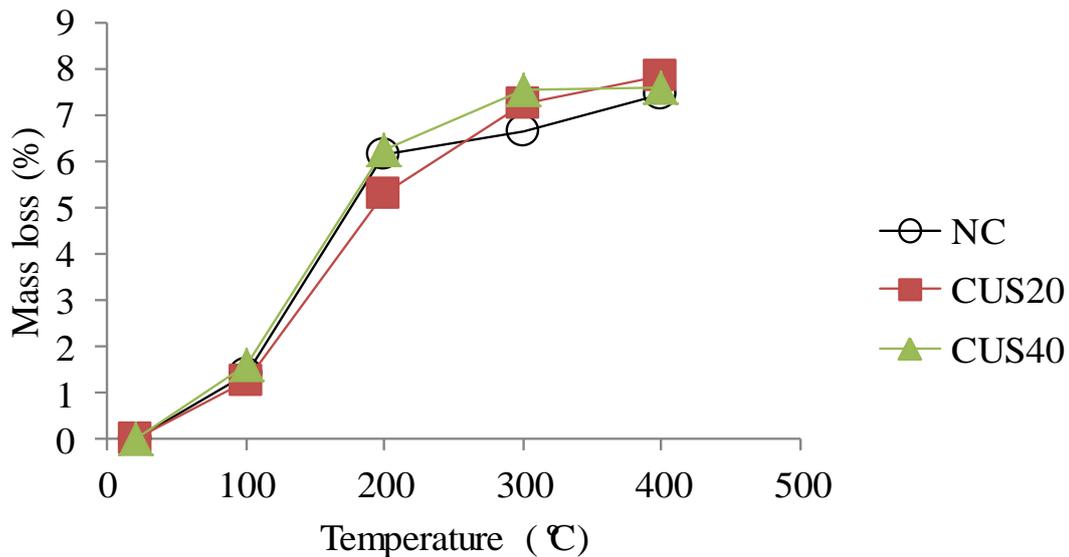


Figure 4.1 Mass loss of concrete after heating.

The mass loss data of the concrete specimens due to the heating are shown in Figure 4.1. It can be seen from the figure that the mass loss data of all kinds of concrete are significantly increasing from 100 °C to 200 °C due to the evaporation of free water contained in the capillary pores. After that, from 200 °C to 400 °C, the mass loss data of all kind of concrete are slightly increased compared with the cases of before 200 °C, the mass loss in this process is mainly caused by the loss of chemically bounded water in concrete. In addition, the influence of copper slag fine aggregate on the mass loss of the high temperature damaged self-compacting concrete is not obvious.

#### 4.3.2 Porosity and SEM observation of micro-structure

The variation curves of the concrete porosity with the heating temperature are shown in Figure 4.2. It can be seen from the figure that the porosity values of all specimens show the similar result with the mass loss. The porosity significantly increasing from 20 °C to 200 °C, which would be due to the developing of the micro-cracks and voids in concrete caused by

the high vapor pressure of the evaporation of free water in the concrete pores. However, from 200 °C to 400 °C, the variations of the porosity become relatively small compared with the cases of before 200 °C for all specimens after the free water in concrete was vaporized.

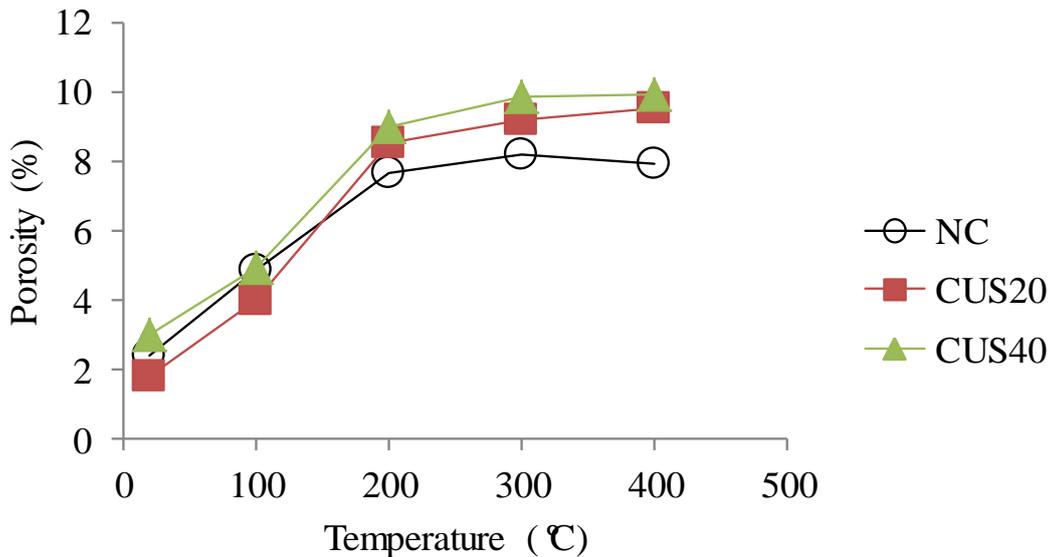


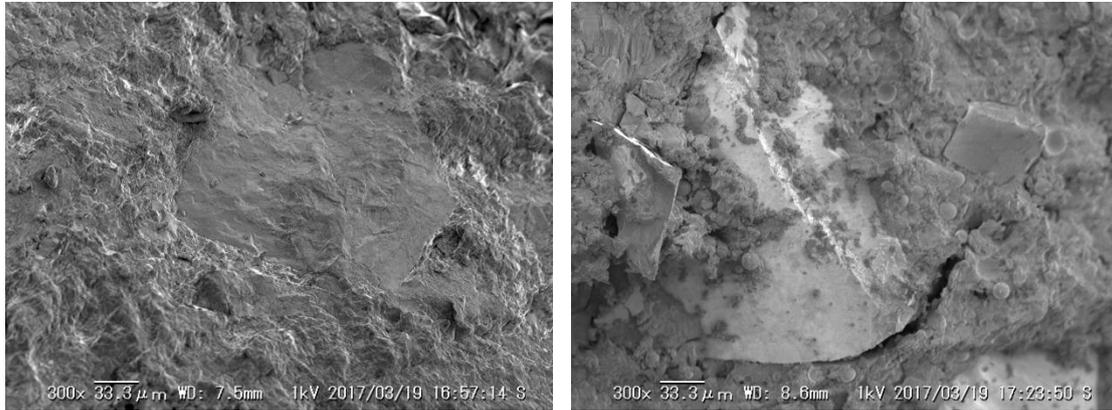
Figure 4.2 Porosity of concrete after heating.

On the other hand, the influence of copper slag on porosity is not obvious when the heating temperature is not exceed 200 °C and the porosity increases with the increasing of the copper slag fine aggregate content in concrete when the heating temperature exceeds 200 °C. The reason of this would be that the relatively high thermal expansion rate of copper slag aggregate might be introduced more micro-cracks and voids around the copper slag aggregate than the case of the normal aggregate concrete (NC).

The SEM photos of the micro-structure around the fine aggregates in concrete under ambient temperature and after heating to 400 °C are shown in Figure 4.3 and Figure 4.4 respectively. The concrete samples for the SEM observation were cut out from the center part of the specimen and the observation magnification of the SEM was set to 300x.

Figure 4.3 (b) clearly shows the micro-cracks and voids between the copper slag aggregate and cement paste compared with the case of normal aggregate (Figure 4.3 (a)). These micro-cracks and voids generated by the

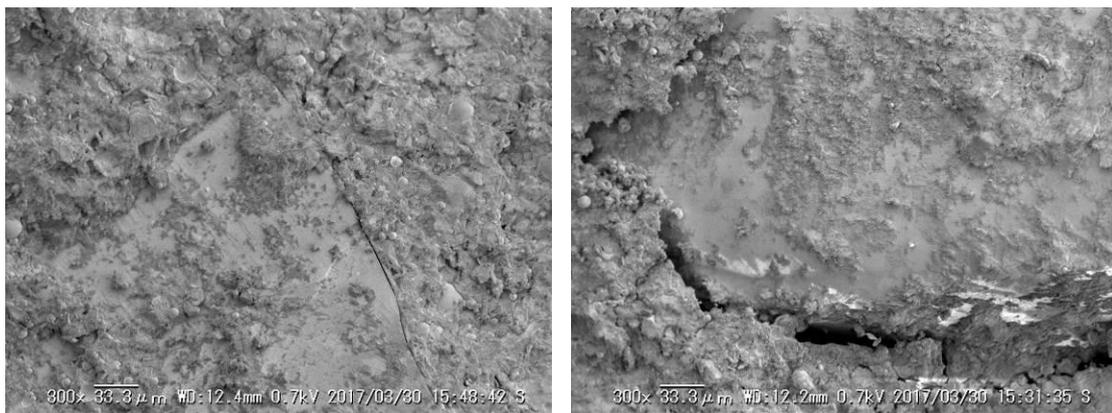
bleeding of copper slag will release the evaporation pressure and reduce the susceptibility of explosive spalling when the concrete is subjected to a high temperature environment.



(a) Normal aggregate

(b) Copper slag aggregate

Figure 4.3 SEM photos of fine aggregates under ambient temperature.



(a) Normal aggregate

(b) Copper slag aggregate

Figure 4.4 SEM photos of fine aggregates after heating to 400 °C.

After heating to 400 °C, as for case of the normal fine aggregate, Figure 4.4 (a) shows the small micro-crack along the normal fine aggregate, and as for the case of copper slag fine aggregate, Figure 4.4 (b) clearly shows the remarkable development of micro-cracks and voids around the copper slag fine aggregate. The reason is considered that the copper slag has the higher thermal expansion rate compared with the case of normal fine aggregate, which will generate the larger thermal expansion of the copper slag fine aggregate than that of the case of normal fine aggregate.

#### 4.3.3 Residual compressive strength

The variation curves of the residual compressive strength of the

self-compacting concrete after heating with the heating temperature are shown in Figure 4.5. It can be seen from this figure that the residual compressive strength of the self-compacting concrete decreases a little with the increase of the copper slag fine aggregate content in concrete. The reason of this compressive strength reduction is considered that the bond strength between the copper slag fine aggregate and the cement paste is less than the case of normal fine aggregate because of the bleeding water around the copper slag fine aggregate surface and the glassy smooth surface of the copper slag fine aggregate. However, from NC to CUS40 self-compacting concrete under ambient temperature, the compressive strength only decreases about 14%, which indicates that such reduction of the compressive strength due to the using of copper slag fine aggregate does not serious when the replacement rate is less than 40% as shown in Figure 4.5.

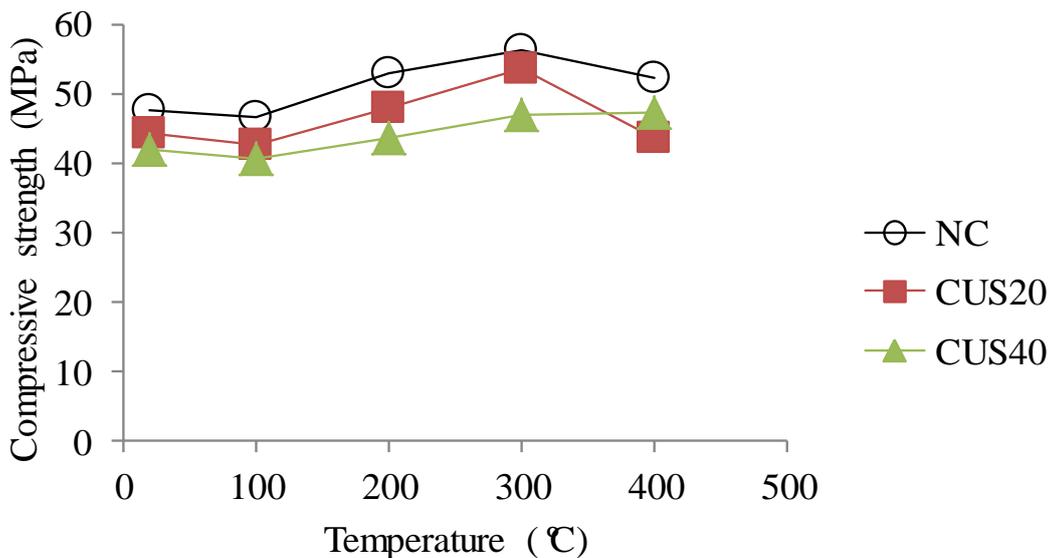


Figure 4.5 Residual compressive strength of concrete after heating.

In addition, the residual compressive strength value of the self-compacting concrete after heating slightly increases for all kinds of concrete specimens. It has been reported in the previous researches that 500 °C is a critical temperature for self-compacting concrete exposed to fire and the reduction of the compressive strength is not critical before this temperature [6-7]. According to these previous related papers, the possible

reason is explained as the re-hydration of the cement paste due to the steam curing effect in concrete [8-9]. Since the self-compacting concrete contains much amount of binders than that of the normal concrete, the re-hydration effect could become significant.

#### 4.3.4 Elastic modulus

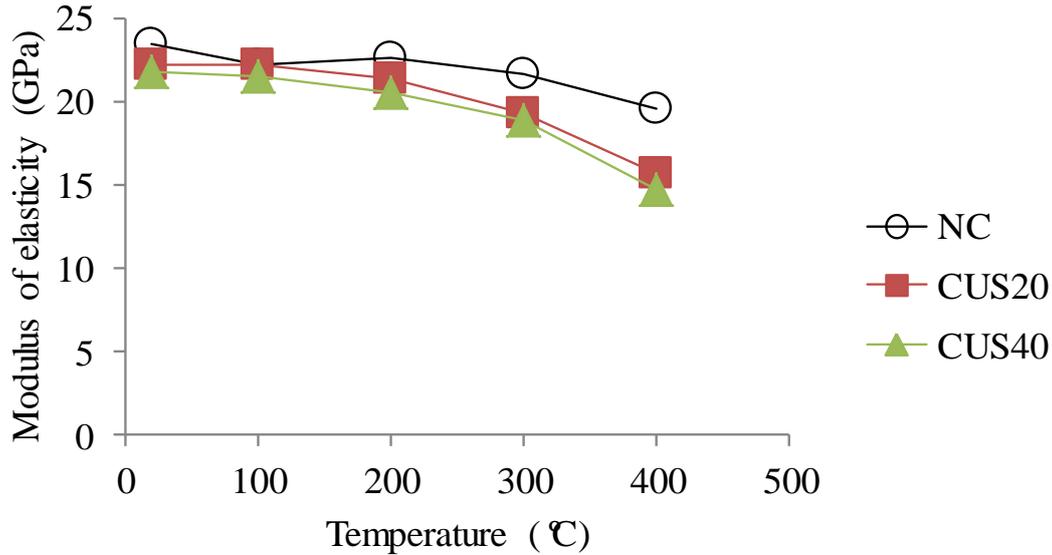


Figure 4.6 Residual elastic modulus of concrete after heating.

Figure 4.6 shows the variation curves of the residual elastic modulus of the self-compacting concrete after heating with the heating temperature. It can be seen from Figure 4.6 that the residual elastic modulus of all kinds of concrete slightly decrease with the heating until 200 °C and such reduction is accelerated from 200 °C to 400 °C especially in the cases of the concrete containing the copper slag fine aggregate (CUS20 and CUS40). Such reduction of the residual elastic modulus would be caused by the micro-cracks or micro-voids introduced at the interface between the fine aggregate and the cement paste due to the thermal expansion during the heating. The thermal expansion rate of the copper slag is higher than that of the normal fine aggregate which means that the micro-crack developing situation of the CUS20 and CUS40 concrete is severer than the case of NC concrete. This will eventually cause the larger decrease of residual elastic modulus of the self-compacting concrete containing copper slag fine aggregate and the result is also consistent with the variation of porosity as

shown in 4.3.2.

On the other hand, the influence of these micro-cracks and micro-voids on the residual compressive strength of the self-compacting concrete was not emerged (Figure 4.5) although the porosity increased due to these micro-cracks and micro-voids at the interface between the fine aggregate and the cement paste generated by the heating (Figure 4.2). Besides the re-hydration reaction of the cement paste in concrete due to the steam curing effect as explained in 4.3.2, another reason is considered that the self-compacting concrete contains relatively high volume binder and low volume aggregate which will promote the pozzolanic reaction. However, such defects could directly affect the deformation performance or the tensile strength of the concrete. So, the residual elastic modulus would decrease with the increase of the heating temperature more sensitively than the case of the residual compressive strength. Such results were also consistent with the previous research [10].

#### 4.3.5 Stress-strain curve of concrete after the heating

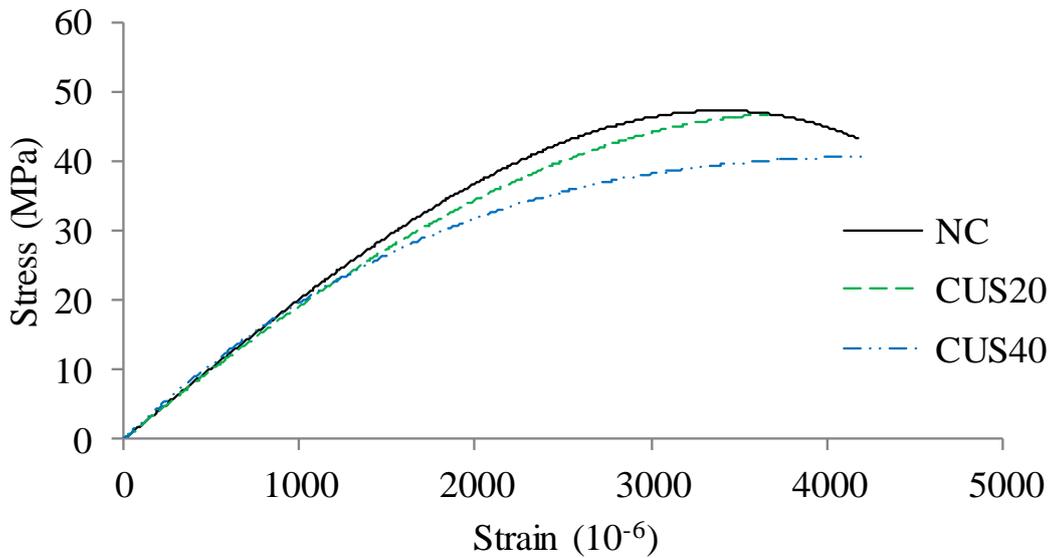
The stress-strain curves of the self-compacting concrete after the heating experiment are shown in Figure 4.7. The comparisons of the stress-strain curves of un-heated NC, CUS20 and CUS40 specimens are shown in Figure 4.7 (a) and the stress-strain curves of high temperature damaged NC, CUS20 and CUS40 concrete are shown in Figure 4.7 (b)~(d) respectively.

It can be seen from Figure 4.7 that at ambient temperature (Figure 4.7 (a)), the influence of the copper slag fine aggregate on the linear part (until 40% of its maximum stress) of the stress-strain curves is not remarkable. However, as for the non-linear part (between 40%~100% of its maximum stress), the stress-strain curve becomes flatter with the increase of copper slag fine aggregate content, which may consistent with the reduction of the compressive strength (Figure 4.5) due to the development of micro-cracks and voids between the interfaces of copper slag fine aggregate and the cement paste which are caused by the bleeding of copper slag aggregate.

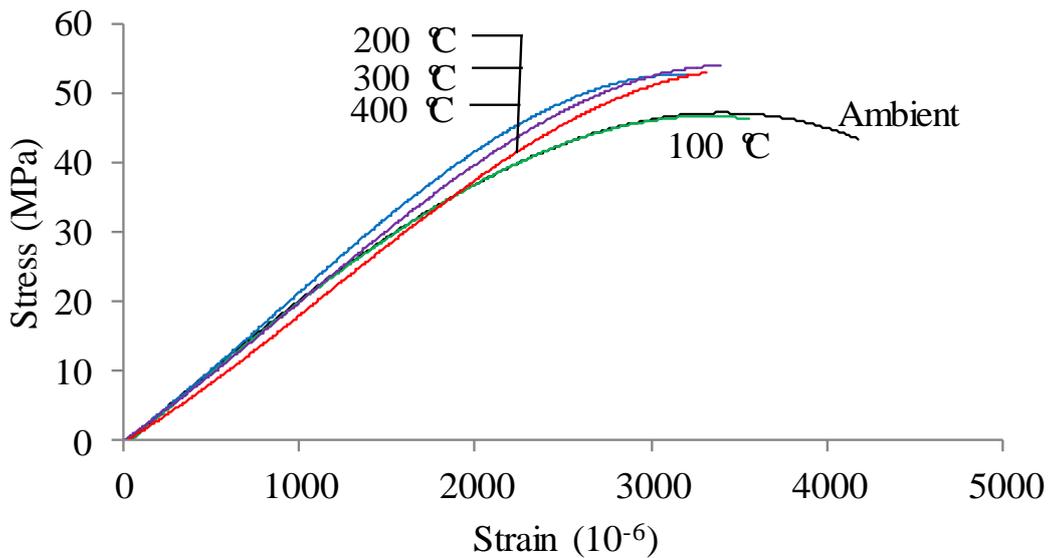
On the other hand, the linear parts of the residual stress-strain curves of NC concrete specimens are extend to the higher stress level (Figure 4.7 (b)) due to the heating. The reason is considered may be caused by the re-hydration in concrete due to the steam curing effect.

However, in the cases of CUS20 and CUS40 concrete specimens, the inclination values of the linear part of the stress-strain curves become small

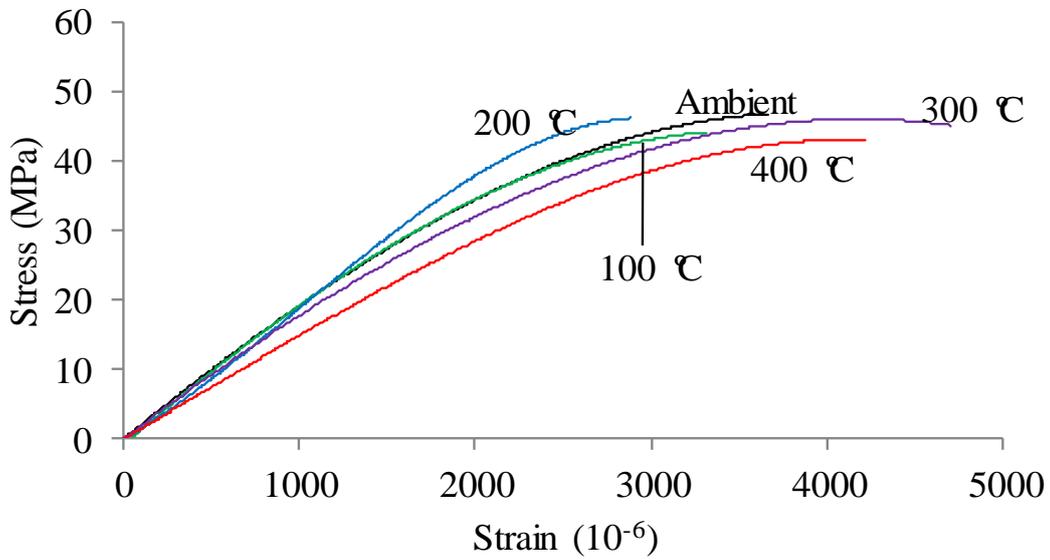
(Figure 4.7 (c) and Figure 4.7 (d)) due to the heating higher than 300 °C, which means the reduction of the residual elastic modulus caused by the development of the micro-cracks and voids introduced at the interfaces between the copper slag fine aggregate and the cement paste. The development of the micro-cracks and voids is promoted by the larger thermal expansion of copper slag fine aggregate.



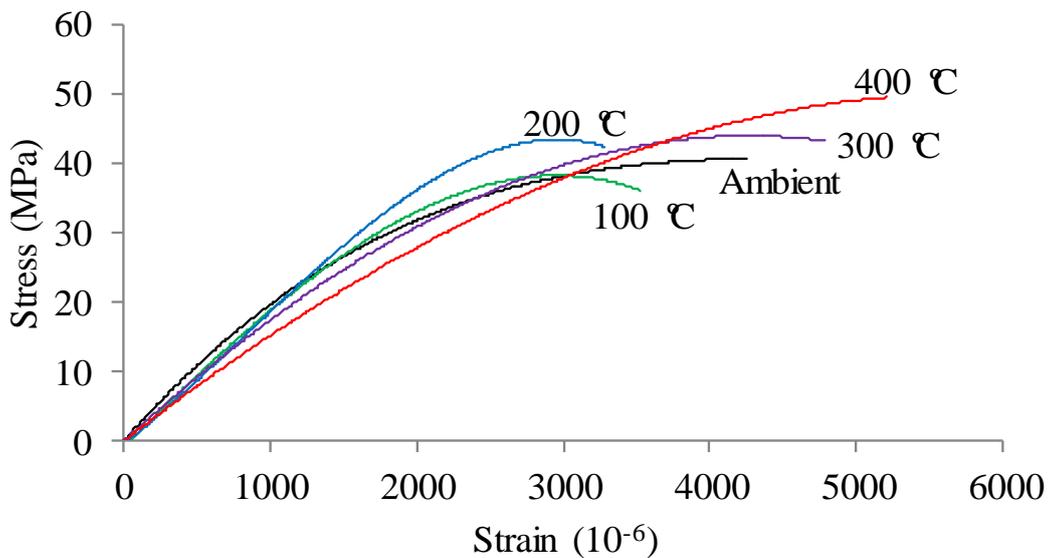
(a) Ambient temperature



(b) NC



(c) CUS20



(d) CUS40

Figure 4.7 Stress-strain curves of concrete after heating.

In addition, the strain values of the cases of CUS20 and CUS40 are extending without the early drop of the stress as the increase of the heating temperature, which indicates that the micro-cracks and voids introduced by the heating do not seriously affect the compressive strength of the self-compacting concrete.

#### 4.3.6 Splitting tensile strength of concrete after heating

The variation curves of residual splitting tensile strength of the self-compacting concrete after heating with the heating temperature are shown in Figure 4.8.

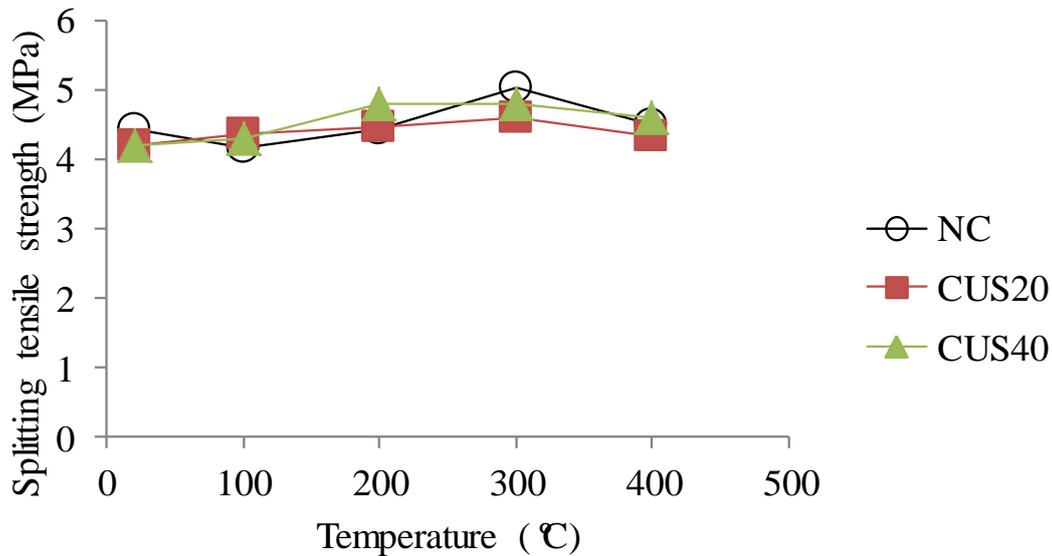


Figure 4.8 Residual splitting tensile strength of concrete after heating.

According to this figure, almost constant values of the residual splitting tensile strength are shown regardless of the kind of concrete and the heating temperature. It is considered that the splitting tensile strength will be more sensitive to the interface between the aggregate and the cement paste than the case of the compressive strength although it will be less sensitive to the interface than the case of elastic modulus. So, this may be the reason why the effect of the heating temperature on the splitting tensile strength is not significant. In addition, the using of fly ash into concrete also could increase the splitting tensile strength of the concrete due to the pozzolanic reactions of the fly ash and similar result was also reported in the previous related research [10].

#### 4.3.7 Cl<sup>-</sup> penetration into concrete after heating

The results of spraying 0.1 mol/L AgNO<sub>3</sub> solution to the split concrete surfaces after the salt water immersion of the heated specimens and the measured Cl<sup>-</sup> penetration depths are shown in Figure 4.9 and Figure 4.10 respectively.

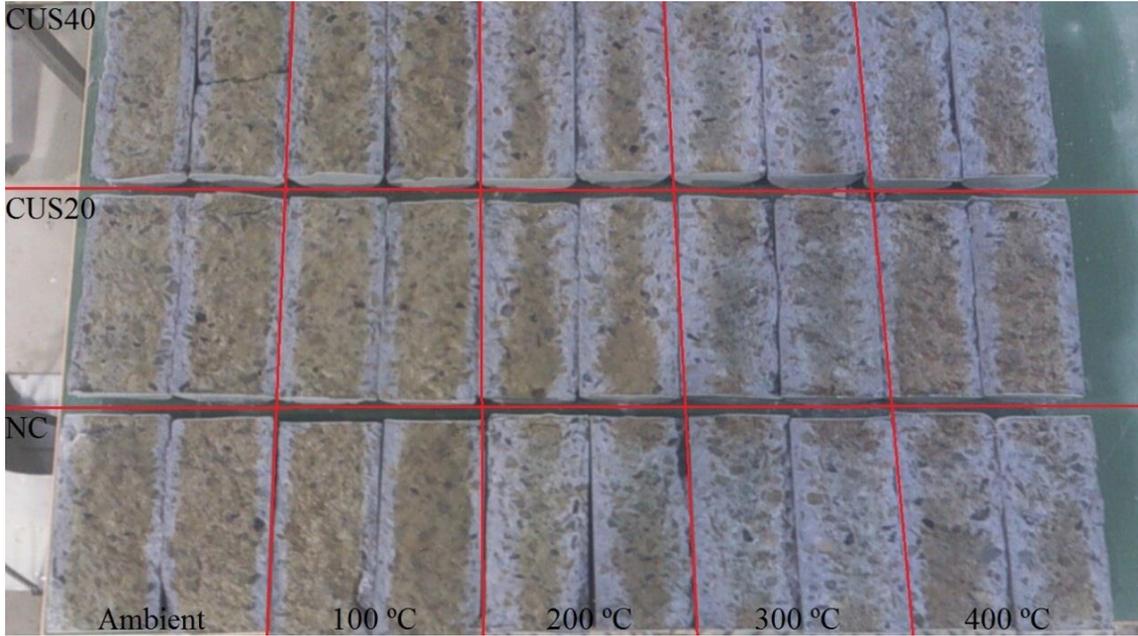


Figure 4.9 Cl<sup>-</sup> penetration situation after spraying AgNO<sub>3</sub> solution.

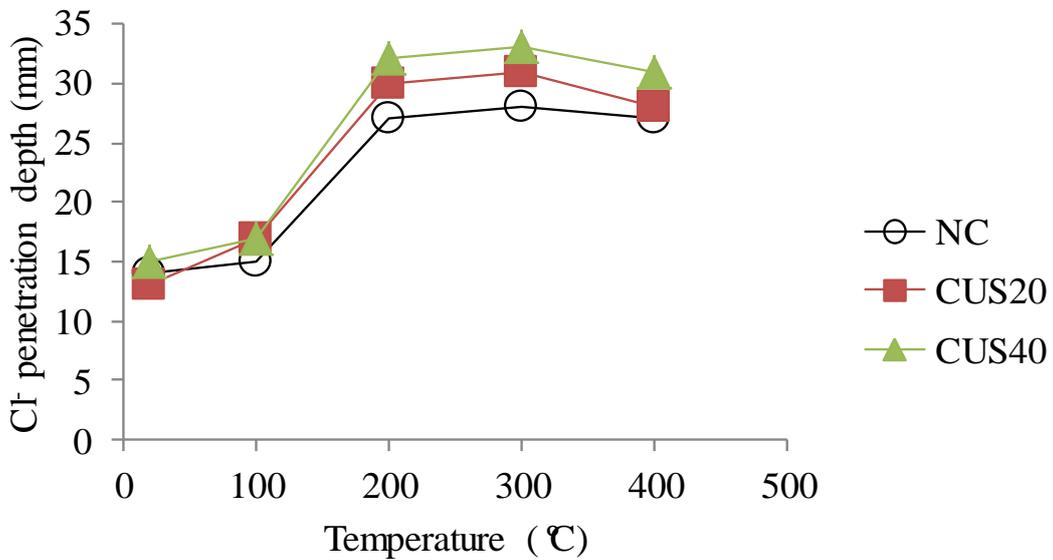


Figure 4.10 Cl<sup>-</sup> penetration depth of concrete.

Figure 4.9 clearly shows the Cl<sup>-</sup> penetration situation of the high temperature damaged self-compacting concrete specimens. It also can be seen from Figure 4.10 that the Cl<sup>-</sup> penetration depth values significantly increase with the rise in temperature from 100 °C to 200 °C and also increased with the increase of the copper slag fine aggregate content when

the heating temperature exceeds 200 °C. Such a tendency of the Cl<sup>-</sup> penetration into concrete after heating is consistent with the results of the concrete porosity variation as shown in Figure 4.2.

In addition, the heating temperature over 100 °C would cause the development of micro-cracks and micro-voids in concrete due to the evaporation pressure of the free water and the mixing of the copper slag aggregate could accelerate the developing of micro-cracks at the interfaces between copper slag fine aggregate and cement paste due to the larger thermal expansion of copper slag. Such micro-cracks and micro-voids could promote the Cl<sup>-</sup> penetration into the self-compacting concrete. From these results, it was found that the heating around 200 °C could damage the durability of the concrete against chloride attack even if the compressive strength didn't decrease.

#### 4.4 Summary

The experimental results of this chapter can be summarized as follows:

(1) Mixing of the copper slag aggregate into self-compacting concrete could reduce the susceptibility of explosive spalling. The compressive strength also decreased with the increase of the copper slag aggregate content.

(2) The residual compressive strength of all kinds of concrete increased after heating up to 300 °C.

(3) More micro-cracks were developed around the interfaces between the copper slag aggregate and the cement paste when the heating temperature exceeded 200 °C compared with the case of the normal aggregate concrete.

(4) The residual elastic modulus of all kinds of concrete decreased when the heating temperature exceeded 200 °C. Especially, the reduction rate of the residual elastic modulus of the concrete containing copper slag aggregate was larger than the case of the normal aggregate concrete.

(5) The heating over 200 °C accelerated the Cl<sup>-</sup> penetration into the concrete and the mixing of the copper slag aggregate also promoted the Cl<sup>-</sup> penetration, which is consistent with the results of the porosity of the heated concrete.

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## Chapter 5 Conclusion

### 5.1 Conclusions of research

Based on the experiments and comparative analysis conducted in this dissertation, the main conclusions can be summarized as follows:

(1) The residual compressive strength deterioration of high temperature damaged concrete was not severe if the heating temperature did not exceed 300 °C. As for the case of self-compacting concrete, the residual compressive strength was even increased due to the re-hydration of the cement paste caused by the steam curing effect if the heating temperature was lower than 400 °C. For the same reason, the compressive strength deterioration degree of higher W/C concrete was severer than that of the lower W/C concrete after high temperature.

(2) The porosity of concrete would be significantly increased when the heating temperature reached 200 °C, which means that the micro-structure of the concrete will be severely destroyed even the deterioration of the residual compressive strength is not obvious if the heating temperature is not too high.

(3) The Cl<sup>-</sup> penetration resistance performance of concrete would be severely damaged due to the development and expansion of micro-cracks in concrete if the heating temperature exceeded 200 °C, which will eventually cause the serious steel corrosion in concrete.

(4) Mixing of copper slag fine aggregate into concrete can obviously improve the fluidity of the fresh concrete due to the glassy smooth surface of the copper slag.

(5) The using of copper slag fine aggregate also reduced the compressive strength of the concrete. Besides the glassy smooth surface of the copper slag, another reason is considered that the copper slag can promote the bleeding of the fresh concrete and the bleeding water will generate micro-cracks and voids in concrete.

(6) As for the self-compacting concrete, the sensitivity of residual splitting tensile strength to the copper slag fine aggregate was higher than that of the residual compressive strength, whereas was lower than the case of residual elastic modulus.

(7) The Cl<sup>-</sup> penetration resistance performance of the high temperature damaged concrete significantly decreased as the increase of heating temperature. Besides the developing of the micro-cracks in concrete, the Cl<sup>-</sup>

fixing ability of the cement paste was also significantly decreased with the increase of the heating temperature, which will accelerate the penetration of Cl<sup>-</sup> into fire damaged concrete.

(8) The steel bar embedded in the 200 and 300 °C concrete specimens was severely corroded compared with that of the un-heated and 100 °C heated concrete specimens although the deterioration of compressive strength was not obvious, which indicates that the residual durability properties of high temperature damaged concrete could be severely deteriorated even the residual mechanical properties does not significantly decrease. Thus, the residual durability properties of high temperature damaged concrete must be considered in the strengthening and rehabilitation engineering for the fire damaged concrete constructions.

## 5.2 Future works

According to the conclusions and the discussions of this dissertation, the following works should also be considered in the future researches:

(1) Different kinds of fibres are often used in concrete to prevent the explosive spalling especially for the high-density concrete. The influences of fibre on the residual properties especially for the Cl<sup>-</sup> penetration and steel corrosion properties of high temperature damaged concrete are still need to be clarified in the future.

(2) The heating temperature in this program is not very high and the deterioration of residual compressive strength is not significant. In order to clearly understand the deterioration process of high temperature damaged concrete, the heating temperature should be increased in the future researches.

(3) Normally, the fire damaged concrete structures will be strengthened and rehabilitated if the heating temperature is not too high. One effective method is to coat the concrete surface using various kinds of antirust coatings, the influences of coating situations on the residual durability performance of high temperature damaged concrete structures are also important to investigate.

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Wei Gong  
July, 2017  
In Tokushima, Japan

## Publications during doctoral course

- [1] Wei Gong, Takao Ueda. Basic Study on Chloride-induced Steel Corrosion in Concrete Subjected to Heating up to 300 °C [J], Journal of the Society of Materials Science, Japan, 2018. (Accepted)
- [2] Wei Gong, Takao Ueda. Properties of self-compacting concrete containing copper slag aggregate after heating up to 400 °C [J], Structural Concrete, 2018. (Accepted)