Scientific paper

Performance Recovery of Fire-damaged Concrete by Impregnation of NaOH-added Lithium Silicate

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Abstract

When concrete is subjected to the elevated temperatures of a fire, cracking occurs and the mechanical performance and durability deteriorate. Current crack repair methods using epoxy or polymer cement cannot repair microscopic damage and cannot restore the performances of whole concrete. There is no effective method to promote the recovery of concrete properties after fire. In this study, the authors proposed to use a highly permeable and alkaline silicate surface impregnation solution (NW-LS) to repair fire-damaged concrete, which is composed of sodium hydroxide (NaOH), lithium silicate (LS) and water and can penetrate in the inside of concrete. The concretes used in the experiments had the compressive strengths of 24.4 to 60.0 MPa before heating, and were heated at different temperatures (300, 500, 650°C) and then cooled in water or air. The changes in the performances, chemical compositions, and internal structure of the concretes after repairing were investigated in detail. The experimental results indicate that the repair densified the internal structure of heated concretes by the reaction between the silicate of the solution and the $Ca(OH)_2$ of the heated concretes, and greatly increased the compressive strength, carbonation resistance, and freezing-thawing resistance of the heated concretes. In addition, the alkalinity of heated concrete was restored by the NW-LS solution.

1. Introduction

Fires occur frequently all around the world, for example, in Japan in 2023, there were 38659 fires, of which 20968 were building fires (FDMA 2023). The susceptibility of wood structures to fire is undisputed, and even concrete structures are subjected to varying degrees of degradation and even collapse. Therefore, the safety of buildings during fire and the restoration after fire are very important issues. Concrete undergoes chemical transformation of cement hydrates at elevated temperature. At heating temperatures of 70-100°C, ettringite disappears, $Ca(OH)_2$ decomposes at 450-550°C, $CaCO_3$ decomposes above 600°C, and at 600-800°C, C-S-H gels decompose to form β-C_2S (Alonso and Fernandez-Municio 2004; Castellote *et al.* 2004; Hager 2013), which cause the hardened cement paste matrix to shrink. The shrinkage of the cement paste and the thermal expansion of siliceous aggregates or the decarbonation of limestone aggregates due to high temperature result in cracking and degradation of mechanical performance and durability of the concrete (Tokuda 1984).

The residual compressive strength of concrete is approximately 75%, 45%, 15% of the original compressive strength after heating at 300°C, 500°C, and 800°C, respectively (AIJ 2017). Mineral admixtures increase heating-induced strength reduction of concrete (Li *et al.* 2012). Besides heating temperature, the significant factors resulting in the performance degradation of concrete include the strength grade of concrete before heating, raw materials of concrete, and cooling method after heating (Furumura 1970; Abe *et al.* 1999). As the effects of cooling method, it has been reported that the compressive strength of concrete rapidly cooled in water is lower than that of concrete naturally cooled in air after heating (Kouchi and Kurihara 2011; Li *et al.* 2016a). Water-cooling may help to heal the surface cracks due to more re-hydration later, but rapid cooling widens the cracks and leads to more cracking (Li *et al.* 2015).

Heated concrete not only cracks, but the pores in the cement paste coarsen and the total amount of pores increases. The higher the heating temperature, the larger the size and amount of pores (Ichise and Kawabe 2002; Li *et al.* 2016a). The increase in porosity is generally thought to be due to dehydration or decomposition of hydrates: $Ca(OH)_2$ and C-S-H gels, etc. (Li *et al.* 2016b). As pores and cracks increase, the permeability of substances such as carbon dioxide, chloride ions, and water increase, which causes the durability of concrete in terms of salt damage, carbonation, and freeze-thaw resistance to decline. Although the $Ca(OH)_2$, decomposed due to heating, can recover from CaO in a humid environment, making the heated concrete alkaline, the high concentration of CO_2 during the fire and the easy

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diffusion of CO_2 in the concrete after the fire increase the carbonate reaction of CaO or $Ca(OH)_2$, reducing the alkalinity of the heated concrete. Li and Li (2011) measured the carbonation depth of heated concrete after the accelerated carbonation test, and found that the higher the heating temperature, the greater the carbonation depth and the lower the resistance to carbonation. It has also been found that the frost resistance of heated concrete, whether normal or high strength, decreases, and that the higher the heating temperature, the greater the decrease in frost resistance, and rapid cooling of water causes a greater decrease than natural cooling in air (Li and Ryuda 2016b, 2016c). Kakae *et al.* (2017) also reported that cooling method affects the physical properties of concrete after heating. Therefore, the repair of concrete after fire needs to be addressed not only the cracks and strength, but also the durability.

Many studies have confirmed that post-fire-curing in a wet environment would restore the performances of heated concrete such as strength and permeability to a certain extent due to rehydration of decomposed $Ca(OH)_2$ and C-S-H gels (Alonso and Fernandez 2004; Li *et al.* 2016b). The rehydration can partially repair cracks and can reduce the pores generated in heated concrete (Li 2015; Li *et al.* 2016a). Mass, dimensions, and dynamic modulus of elasticity are also somewhat restored by re-curing (Li *et al.* 2015a). Poon *et al.* (2001) reported that the re-curing in water restored the strengths of the concretes with 0.3, and 0.5 of water-cement (WC) ratio, after heating below 600°C. Suzuki *et al.* (2009) reported that after high-strength mortar with 0.3 of W/C was heated at 550°C, its compressive strength continued to decrease when left in the air, but when re-cured in water, the strength recovered up to 80%. It has been also found that the strength and carbonation resistance of heated concrete using either ordinary Portland cement or slag cement recovered to some extent after re-curing, and the lower the heating temperature, the higher the recovery rate, as well as water re-curing led to a higher recovery rate compared with air re-curing (Li and Li 2011). The bond strength between heated concrete and reinforcement and the frost resistance of heater concrete are also restored to some extent by re-curing (Li *et al.* 2015b; Li and Ryuda 2016a, 2016b, 2016c).

However, although post-fire curing in a moist environment can restore the properties of concrete to some degree, this restoration is extremely limited when heating temperatures reach 500°C or more since the rehydration of decomposed C-S-H gels is not complete. And it is not practical to supply water to actual concrete structure for more than one month of re-curing. Therefore, AIJ (2024) recommends that when the heating temperature of concrete surface is below 500°C and the concrete still has a compressive strength above its design strength, the cracks are repaired and the surface is coated to prevent further internal degradation, but when the heating temperature exceeds 500°C, the deteriorated concrete needs to be removed and recast if its strength and durability are greatly reduced. However, re-constructing concrete is considerably labor intensive and costly, and repairing surface and cracks does not restore the overall strength and durability of the concrete since internal repair can not be provided.

Silicate-based concrete modifier, e.g., water glass or lithium silicate, which is generally used for lowering the permeability of water and chloride ion, was proposed to repair fire-damaged concrete based on the reaction between the $Ca(OH)_2$ in concrete and silicate (Li and Li 2011, 2013). The experimental results showed that the compressive strength of concrete coated with the concrete modifier increased by 3.8%, 3.7%, 11.0%, 17.3% and 6.1%, respectively, for the heating temperatures of 150, 300, 450, 600 and 750°C, compared to heated/uncoated concrete without coating. The most significant improvement by the coating in compressive strength and carbonation resistance was observed for the concrete heated at 600°C. This is mainly because the higher the heating temperature, the looser the concrete becomes, and the more easily the modifier penetrates in, thus increasing the reaction between silicate and $Ca(OH)_2$. However, at or above 700°C, calcium-silicate-hydrate (C-S-H) gels are decomposed, leading to a significant reduction of concrete strength, even with less than 40% of its original strength, and high-strength concrete may spall during heating (Alhamad *et al.* 2022; Kodur 2014; Bastami *et al.* 2010). Generally, the concrete heated above 700°C can not be repaired by silicate-based modifier due to excessive degradation or even spalling. Hence, the repair utilizing silica-based modifier is mainly for the concrete heated below 700°C, the extents to which performances of heated concrete are restored, however, depend on the penetration of modifier.

The authors found accidentally that the permeability of lithium silicate solution, one of conventional silicate-based surface impregnation materials or modifiers, can be greatly improved by adding sodium hydroxide (NaOH) solution (Li 2023). Since the solubility of $Ca(OH)_2$ in aqueous NaOH solutions decreases (Pallagi *et al.* 2012), we estimate that the addition of NaOH delays the reaction between the $Ca(OH)_2$ in concrete and lithium silicate (LS), thus LS solution can penetrate deeper into concrete. Our recent detailed investigation confirmed that the addition of NaOH and water to LS makes the blending solution, abbreviated as NW-LS, have excellent permeability when the Na/Si molar ratio falls into the range of 0.5 to 1.25 and the solid concentration is below 17%, no matter for heated and unheated concrete, normal concrete or high strength concrete (Kitada and Li 2024). The NW-LS solution is expected to be used to not only repair internal damage of fire-damaged concrete, but also to re-alkalize the concrete due to the carbonation during firing or post-fire. The chemical realkalization method has been usually used to re-alkalize the neutralized concrete that restores the alkalinity of concrete for protecting steel

Table 1 Mix proportions and mechanical properties of concretes.

Series	W/C(%)	Unit mass (kg/m^3)									Compressive strength (N/mm^2)
		W	C	S-1	S-2	S-3	G-1	G-2	G-3	WR	
C24	68	164	241	276	643	-	1000	-	-	2.7	24.4
C35	56	183	332	-	-	770	-	-	1009	1.66	35.6
C40	53	164	309	254	591	-	1020	-	-	2.97	40.7
C42	53	164	309	265	618	-	-	919	-	4.08	42.7
C60	37	168	454	221	516	-	-	937	-	5.45	60.0

[Notes] W: water, C: cement, S: fine aggregate, G: coarse aggregate, WR: Air entraining and water reducing agent.

Table 2 Raw Materials used in the concretes.

Notation	Raw material	Density (g/cm^3)	Fineness modulus (F.M.), grading, and percentage of absolute volume (PAV)
C	Ordinary Portland cement	3.16	Blaine value: 3500 cm^2/g
S-1	Crushed sand (rhyolite)	2.58	F.M.: 2.9, Grading: 0-5 mm
S-2	Processed sand (granite)	2.55	F.M.: 2.7, Grading: 0-5 mm
S-3	Sea sand (siliceous)	2.57	F.M.: 2.9, Grading: 0-5 mm
G-1	Crushed stone (diabase)	2.81	Grading: 20-5 mm, PAV: 60.6%
G-2	Crushed stone (rhyolite)	2.64	Grading: 20-5 mm, PAV: 58.1%
G-3	Crushed stone (sandstone)	2.73	Grading: 20-5 mm, PAV: 59.2%

reinforcement by introducing alkaline components into the concrete (Réus and Medeiros 2020). Moreover, the water included in the NW-LS solution can contribute to the rehydration of decomposed hydrates of concrete.

In this study, in order to develop effective method for the repair of fire-damaged concrete, we investigated the strength recovery of different strength grades of concrete repaired by impregnation with the NW-LS solution, after heating them at different elevated temperatures and cooled them by different methods. The effects of the NW-LS solution on the recovery of carbonation resistance and frost resistance of heated concrete were also examined. Finally, the changes in chemical compositions and microstructures of fire-damaged concretes after repaired with the NW-LS solution were clarified by XRD and SEM-EDS analyses.

2. Experimental program

2.1 Preparation of concrete specimens

Five strength grades of concrete were used for the restoration experiments in this study, and their mix proportions and raw materials used are shown in **Tables 1** and **2**. Since the concrete specimens were produced at different periods, siliceous aggregates from different manufacturers were used. Thus, even for the same type of aggregate, they had different physical properties due to different original rocks and manufacturers.

The specimens were prepared in accordance with JIS A 1132:2020 (Method of making and curing concrete specimens). The specimens for compressive strength measurements were cylinders with a diameter of 100 mm and a height of 200 mm, while the specimens for the carbonation resistance and freeze-thaw resistance tests were prisms of 100×100×400 mm. After the production, the specimens were demolded 24 hours later, and then cured in a 20±2°C water tank for 28 days. The 28-day compressive strengths are presented in **Table 1**.

The concrete specimens were divided into three groups. Group 1 was used to measure the performances of concretes in the unheated state after the 28-day water curing. Group 2 was used to measure the performances after heating and cooling, but non-repairing. Group 3 was for the performance tests conducting after being heated, cooled, repaired by the NW-LS solution, and re-cured.

The specimens of Groups 2 and 3 were heated using an electric furnace after the 28-day water curing. The heating and cooling regimes are shown in **Fig. 1**. The target heating temperatures were set to be 300°C, 500°C, and 650°C, and

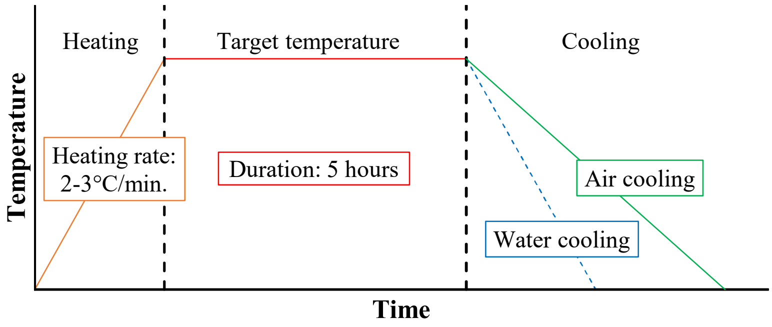


Fig. 1 Heating and cooling regime.

were maintained for 5 hours. To prevent the specimens from spalling due to rapid heating, the heating rate was set as 3.0°C/min. Ichinose *et al.* (2002) reported that the heating rate within the range of 2.5-7.5°C/min. has almost no effect on degradation of compressive strength at elevated temperatures. The heating experiment in the range of 100-700°C at a heating rate of 0.77°C/min. or 1.0°C/min., performed by Kim *et al.* (2014), showed that when the target temperature was maintained for 60 minutes, the difference in the internal and external temperatures of cylindrical specimen of 100 mm in diameter and 200 mm in height was within 5°C. Thus, although we did not measure the internal temperatures of the specimens, long-time (5 hours) maintenance of the target temperature allowed the internal temperatures of specimens with 100 mm of diameter or of cross-sectional side to reach the temperature of the electric furnace.

Air cooling method was used for all the concrete series after being heated. However, for Series C24, water cooling method was also used to investigate the effect of cooling method on the recovery of mechanical properties of heated concrete after the restoration using the NW-LS solution. For the natural air cooling, the specimens were left to place in the electric furnace until they reached room temperature, then they were removed from the electric furnace and stored for 7 days in a room with 20±3°C and 60±5% RH. For the water cooling, the specimens were removed from the electric furnace immediately after heating and then sprayed with tap water for 30 minutes. After the water spraying, the specimens were stored for 7 days in the room, where the air-cooled specimens were kept, to make them have the same water content with the air-cooled specimens.

After 7 days of storage, the specimens of Group 3 were immersed in the NW-LS solution for 3 days to ensure full penetration of the solution. Our past immersion experiment have already confirmed that the NW-LS solution can penetrate to the center of the heated specimen after 3-day immersion (Kitada and Li 2024).

The NW-LS solution used was prepared by blending the commercially available lithium silicate (SiO_2: 21%, Li_2O: 2.9%, SiO_2/Li_2O: 3.6, pH: 10.0-11.4, density: 1.20 g/cm^3, viscosity at 25°C: 13 mPa·s), NaOH aqueous solution (concentration: 10 mol/L, density: 1.33 g/cm^3, mass concentration 32%), and deionized water in a volume ratio of 2:1:2. Thus, the W-LS solution had 1.25 of Na/Si molar ratio, and 16.6% of solid concentration.

2.2 Measurement of properties before and after repairing

2.2.1 Mechanical property tests

The compressive tests were performed for the three series of concretes: C24, C42, and C60, according to JIS A 1108:2018 (Method of test for compressive strength of concrete), before and after heating at 300°C, 500°C and 650°C, and after repairing with the NW-LS solution. Groups 2 and 3 were measured at 7 and 38 days after heating, respectively. The compressive strengths were mean values of three cylinders.

2.2.2 Accelerated carbonation resistance test

100×100×400 mm Group 2 and Group 3 specimens of Series C24 and Series C40, which were heated and then cooled naturally in the air, were used for the accelerated carbonation testing. The heating temperatures were 500°C and 650°C. The accelerated carbonation resistance test was carried out in the chamber at 20°C, 60% RH, under CO_2 concentration of 5% for 31 days (3-day immersion in the NW-LS solution and 28-day re-curing in air) for Group 3, and for 7 days (stored in air) for Group 2. respectively, after the heating. Group 2 specimens used were cooled in air and then stored in the room with 20±3°C and 60±5% RH for 7 days. The specimens of Group 3 were re-cured in the same room. Therefore, the moisture contents of the repaired and non-repaired specimens were the same from the beginning of the accelerated carbonation test.

During the accelerated carbonation test, the test specimens were taken out from the chamber every 7 days and cut with a concrete cutter, then the cut section was sprayed with 1% phenolphthalein solution. The coloration of the cut surface was photographed, and carbonation depth was measured. The carbonation depth was an average of four points on each side of the cut surface (total 8 locations). After measurement, the cut surface was sealed with impermeable tape to prevent CO_2 penetration from the cut surface, and then the specimen was returned to the chamber.

2.2.3 Freezing-thawing resistance test

100×100×400 mm Group 2 and Group 3 specimens of Series C35, which were cooled in the air after heating, were used for freezing-thawing resistance testing. The heating temperatures were 500°C and 650°C. The non-repaired specimen was stored in the room with 20±3°C and 60±5% RH for 31 days after heating. At 31-day age after the heating, the specimens were soaked in water for two days to saturate them, their initial mass and the initial relative dynamic modulus were measured.

The freezing-thawing resistance test was conducted on the repaired and no-repaired specimens at the same age in accordance with JIS A 1148 (Method A of test for resistance of concrete to freezing and thawing in water). The central temperature of the specimen was changed from 5°C to −18°C, then return to 5°C. A freezing-thawing cycle took 3 to 4 hours. The relative dynamic modulus and mass change ratio were measured respectively at every 30 cycles until the specimen was broken and the measurement of relative dynamic modulus could not be done.

3. Experimental results and discussion

3.1 Mechanical properties after heating and after repairing

Table 3 and **Fig. 2** show the residual compressive

Table 3 Residual compressive strength after heating or after heating and repairing.

Series	Heating temperature (°C)	Air-cooling		Water-cooling	
		Non-repaired	Repaired	Non-repaired	Repaired
C24	300	88.4%	113.6%	91.5%	113.8%
	500	56.1%	111.3%	76.8%	99.9%
	650	30.2%	99.0%	58.3%	81.6%
C42	300	82.1%	88.9%	-	-
	500	57.1%	70.2%	-	-
	650	42.2%	73.2%	-	-
C60	300	80.2%	87.4%	-	-
	500	37.2%	83.2%	-	-
	650	31.5%	75.5%	-	-

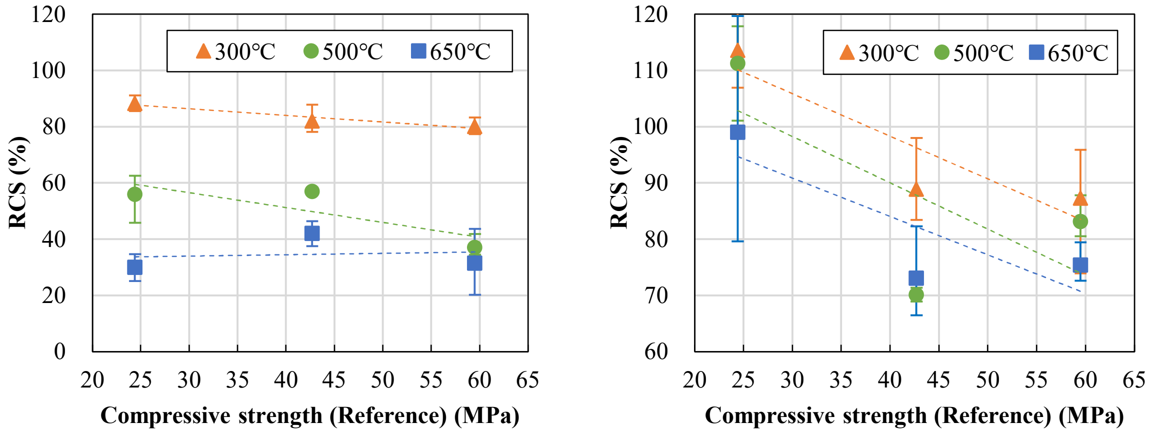
strengths (RCS) of the Group 2 and Group 3's specimens of the three series of concrete (C24, C42, C60), which were measured at 7 days after the specimens were heated and then cooled (Group 2), and after the specimens were heated, cooled, repaired (immersed in the NW-LS solution for 3 days), and re-cured for 28 days (Group 3), respectively. And the effects of heating temperature and cooling method on the RCS are shown in **Fig. 3**.

As shown in **Fig. 2**, the RCS after heating and air cooling (Group 2) was 80%-88% at 300°C, 37%-57% at 500°C, and 30%-42% at 650°C. As shown in **Fig. 3**, the strengths of Series C24 and C42 decreased linearly with heating temperature from 300°C to 650°C, while the strength loss of Series C60 tended to slowly increase from 500°C to 650°C, compared to the sharp increase in the strength loss from 300°C and 500°C. These residual compressive strength ratios were well consistent with general knowledge (AIJ 2017).

The residual compressive strength of all series of concrete, which were repaired with the NW-LS solution (Group 3), increased regardless of heating temperature and cooling method. The specimens with a lower strength grade before heating showed a higher compressive strength recovery. For Series C24, the strengths of the repaired specimens were equal to or higher than the original compressive strength before heating. The residual compressive strength ratios of Series C40 and C60, also reached about 70% or more.

When the cooling methods were compared, we found that the residual compressive strength of Series C24 tended to be higher in case of water cooling, compared to the natural air cooling, as shown in **Fig. 3(a)**. Botte and Caspeele (2017) reported that immediately after heating, rapid cooling by water would result in more cracking and a greater strength loss, compared to natural air cooling, followed by a gradual strength growth due to rehydration reaction because water cooling supplied water. Poon *et al.* (2001) also reported that for ordinary strength concrete with a W/C of 0.50 and a 28-day compressive strength of 35.8 MPa, when heated at 600°C, the compressive strength immediately after heating decreased to 30%, but the strength recovered to 66% by subsequent 56-day curing in water. In the present study, though the water cooling resulted in a larger decrease in strength, compared to the air cooling, but the supply of water during cooling caused an increase in the compressive strength in the specimens of Series 24.

For the Series C24, the compressive strengths of the specimens repaired after the air cooling were higher than those of the specimens repaired after the water cooling.

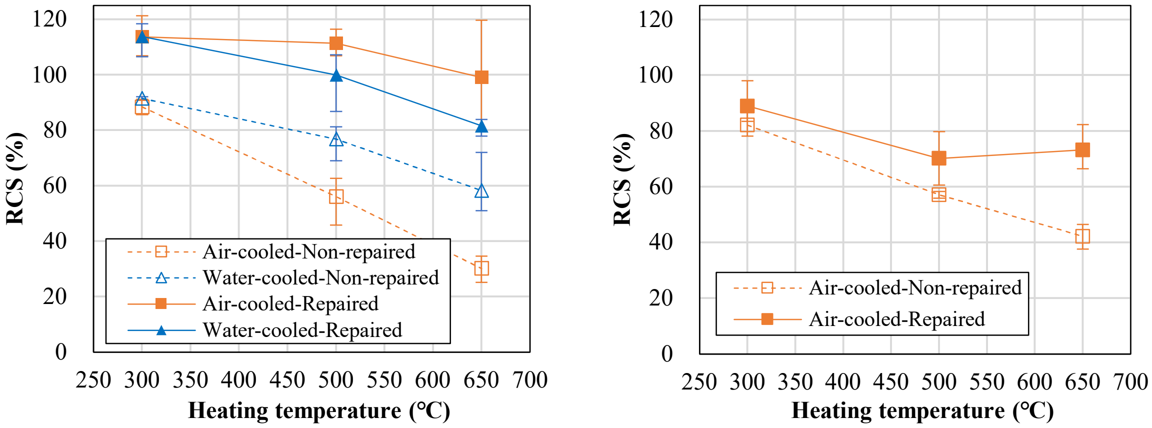


(a) After heating and air cooling

(b) After heating, air cooling and repairing

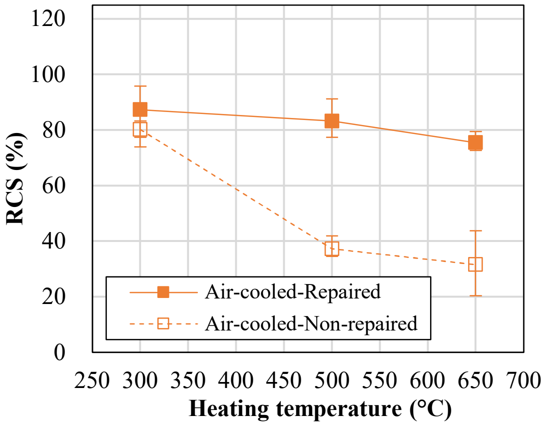
[Note] The bars on the graphs indicate the range of data variation.

Fig. 2 Relationship between residual compressive strength (RCS) and original compressive strength.



(a) Series C24

(b) Series C42



(c) Series C60

[Note] The bars on the graphs indicate the range of data variation.

Fig. 3 Effects of heating, cooling and repairing on the residual compressive strength (RCS) of concrete with different original strengths.

This may be due to the fact that in the air-cooled concrete, the rehydration reaction also occurred due to the water supplied by the NW-LS solution, but the air-cooled specimens had fewer cracks than the water-cooled specimens. Anand *et al.* (2023) and Kim *et al.* (2014) reported that water-cooled concrete exhibits increased cracking, compared to air-cooled concrete, due to the thermal stresses induced by rapid cooling. The NW-LS solution, unlike epoxy-based materials or polymer-cement used as crack fillers, does not solidify and fill cracks by itself. As a result, the NW-LS solution may not be very effective in repairing large cracks.

3.2 Accelerated carbonation resistance

Tables 4 and **5** show the coloration after spraying with the phenolphthalein solution for the C24 and C40 concretes, which were heated at 500°C or 650°C, and then cooled in the air, or/and repaired/re-cured, respectively. Based on tracing the color-changing borders, the carbonation depth of each specimen was measured. The relationships between the carbonation depth and the elapsed time are shown in **Fig. 4** for specimens with repairing or not after heating/air-cooling.

When heated at 500°C, the carbonation depth of the C24 specimen without repairing was 8 mm after 28 days, and 14 mm after 84 days from the beginning of the accelerated carbonation test. On the other hand, for the C24 specimen with repairing, the carbonation depth was 2 mm after 28 days, and 8 mm after 84 days. When heated at 650°C, the carbonation depth of the C24 specimen without repairing reached 33 mm at 28 days, and 42 mm at 35 days from the beginning of the accelerated carbonation test, and thus the measurement was stopped after 35 days because the radius of the specimen was 50 mm. However, for the repaired C24 specimen, the carbonation depth was 12 mm after 28 days, and 16 mm after 35 days. Even after 84 days of carbonation time, the carbonation depth was only 22 mm. From these results, it was found that the carbonation resistance of concrete decreased significantly after heated at the elevated temperatures, especially at 650°C. This is due to the changes of internal structure caused by the

Table 4 Coloration of sliced surface of C24 specimens after spraying with the phenolphthalein solution.

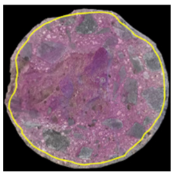
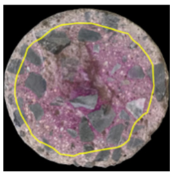
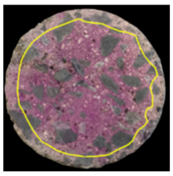
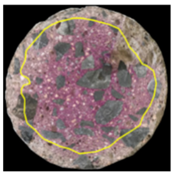
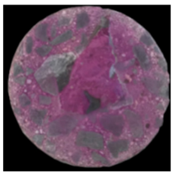
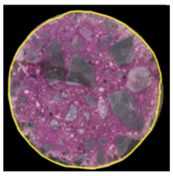
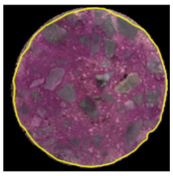
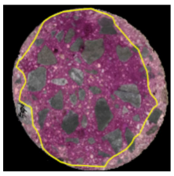
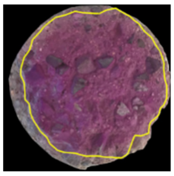
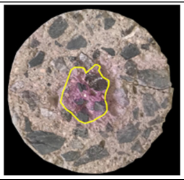
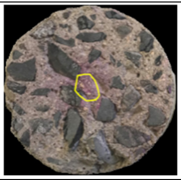
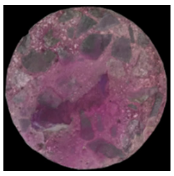
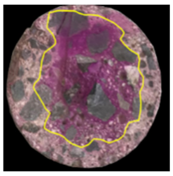
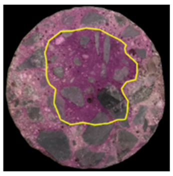
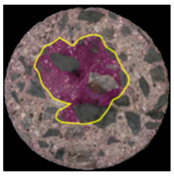
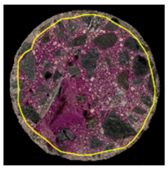
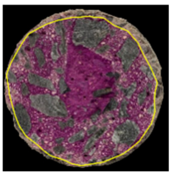
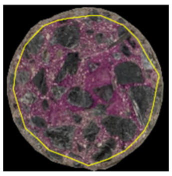
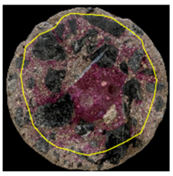
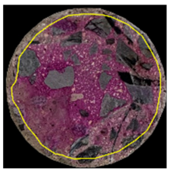
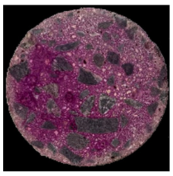
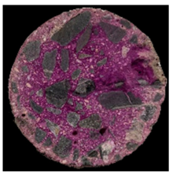
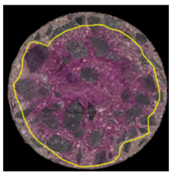
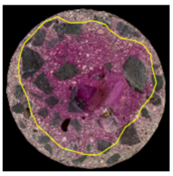
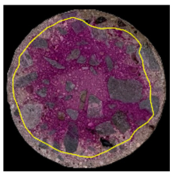
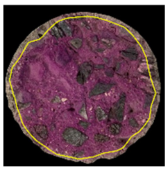
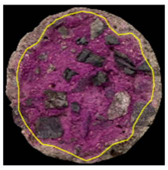
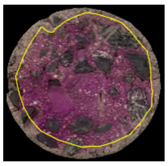
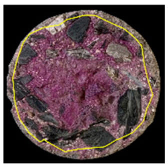
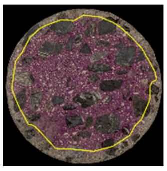
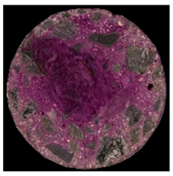
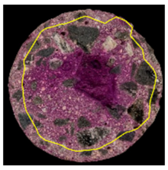
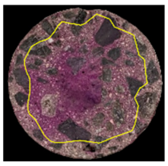
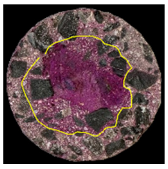
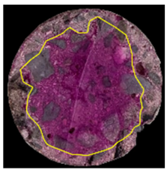
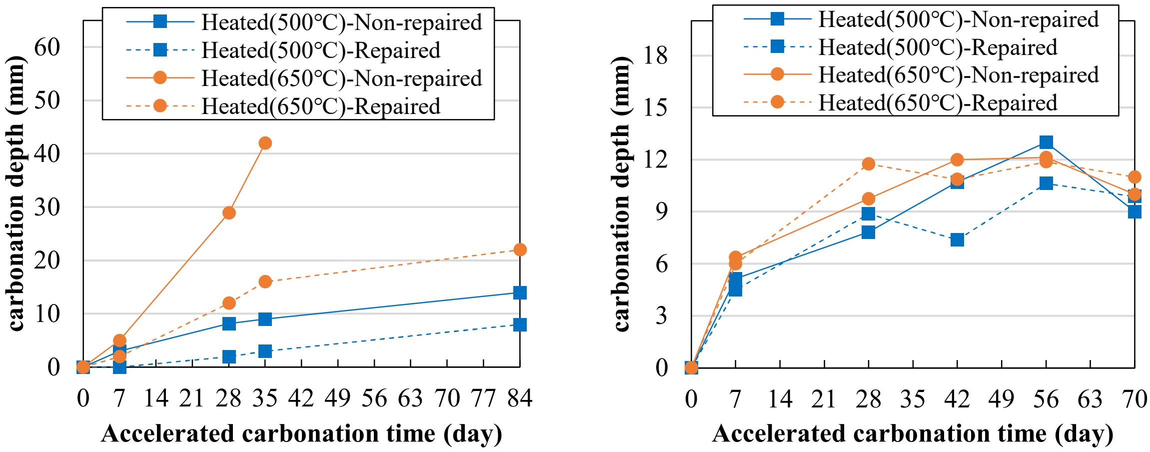
Heating Temperature	Repaired or not	7 days	28 days	35 days	84 days
500°C	Non-repaired				
	Repaired				
650°C	Non-repaired				Not measured
	Repaired				

Table 5 Coloration of sliced surface of C40 specimens after spraying with the phenolphthalein solution for part of carbonation times.

Heating Temperature	Repaired or not	7 days	28 days	42 days	56 days	70 days
500°C	Non-repaired					
	Repaired					
650°C	Non-repaired					
	Repaired					



(a) Series C24 (b) Series C40

Fig. 4 Carbonation depths of non-repaired and repaired concretes.

decomposition of hydrates and the formation of cracks. It was also clearly found that the repairing with the NW-LS solution increased the carbonation resistance of the heated concrete.

On the other hand, as shown in **Table 5** and **Fig. 4(b)**, for the same heating temperature and the same carbonation time, the carbonation depth of the C40 specimen was less than that of the C24 specimen. Feng *et al.* (2023) also found that the pH of the heated concrete during the accelerated carbonation test decreased greatly with the increase of heating temperature, which was mainly because the increase of porosity of the heated concrete promoted the diffusion of CO_2. They also pointed out that the smaller the water-cement ratio, the less the decrease in the pH. That is to say, the loss of carbonation resistance of heated concrete with higher strength is small, which is consistent with the result of this study. In the repaired C24 concretes, the tendency for the depth of carbonation being reduced by NW-LS solution treatment was clearly observed, i.e., the carbonation resistance was improved by repairing. However, in the C40 concrete, little difference in the carbonation depth was observed between the repaired and non-repaired specimens, regardless of the heating temperature. This is because the heated C40 concrete had a small carbonation depth before the repairing, i.e., it still had a relatively high carbonation resistance, so the effect of the repairing was limited. In other words, the NW-LS solution was more effective in improving the carbonation resistance of ordinary strength concrete (<36 MPa) after a fire.

3.3 Freezing-thawing resistance

Figures 5 and **6** shows the measured results of relative dynamic modulus of elasticity and mass loss ratio (positive value: mass decrease, negative value: mass gain) for the C35 specimen after each 30 cycles of freezing-thawing. The non-heated specimen showed a slight decrease in the relative dynamic modulus of elasticity and in mass after 300 cycles. This is because the concrete used the air-entraining water reducer, thus had 4.5% of air content. However, for the specimen heated at 500°C but not repaired, the relative dynamic modulus gradually decreased after about 100 cycles and was halved after 240 cycles, and the mass loss reached 3.6% after 270 cycles. For the specimen heated at 650°C but not repaired, the relative dynamic modulus of elasticity decreased rapidly, even it could not be measured after 30 cycles due to severe damage caused by the frost action. After 60 cycles, the 650°C-heated/non-repaired specimen was broken. That is to say, the frost resistance of concrete was greatly decreased after subjected to an elevated temperature.

On the other hand, for the specimen heated at 500°C and then repaired by the NW-LS solution, the relative dynamic modulus of elasticity remained almost 100% after 300 cycles, which indicates that the 500°C-heated concrete reached the equivalent frost resistance to the unheated concrete after repaired with the NW-LS solution. For the repaired specimen that was heated at 650°C, although its relative dynamic elasticity modulus was lower than 60% after 150 cycles, its frost resistance was greatly increased by the repairing, compared to the non-repaired specimen. Therefore, the application of the NW-LS solution can contribute to improvement of frost resistance of heated concrete.

The mass of the specimen, which was heated at 650°C but non-repaired, gained 0.9% after 30 cycles, though the relative dynamic elasticity modulus was only 33%. The mass gain may be because the mass loss due to surface spalling was smaller than the increase of water absorption resulting from the pore enlargement within the specimen after freezing-thawing cycle. The rehydration of decomposed hydrates may be another reason. As shown in Photo (c-2) of **Table 7**, severe surface spalling did not occur, though wide cracks were found. However, the mass of the 500°C-heated/non-repaired specimen decreased with the freezing-thawing cycle. After 270 cycles, the mass loss reached 3.6%. As shown in Photo (a-2) of **Table 6**, surface spalling resulted in the mass loss.

As shown in **Fig. 6**, we found that the mass of the 500°C-heated/repaired specimen increased with the freezing-thawing recycle, but the 650°C-heated/repaired specimen showed a decline tendency. As shown in Photos (b-2) and (d-2) of **Table 6**, after 270 cycles, the surface degradation of the 500°C-heated/repaired specimen was not observed. Its mass gain was thus because of the water absorption increase, rehydration of decomposed hydrates, and the continuous silicate reaction between LS and Ca^{2+}. However, the surface damage was obviously found from 240 cycles for the 650°C-heated/repaired specimen. By comparing Photos (c-3) and (d-3) in **Table 7**, here is no doubt that the repairing with the NW-LS solution can improve the frost resistance of the heated concrete. According to **Figs. 5** and **6**, when the heating temperature was not above 500°C, the repairing can restore the frost resistance of heated concrete to the original level before heating.

4. XRD and SEM analysis

4.1 Samples and method of analysis

X-ray diffraction (XRD) analysis and Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy (SEM-EDS) analysis were performed to investigate the structural and compositional changes in the C24 and C60 concretes after repairing with the NW-LS solution and re-curing in air. The samples used for the analyses were taken rom cylindrical concrete specimens, and the collection positions are shown in **Fig. 7**. The sampling positions were in the center zone (0-20 mm from the center of the

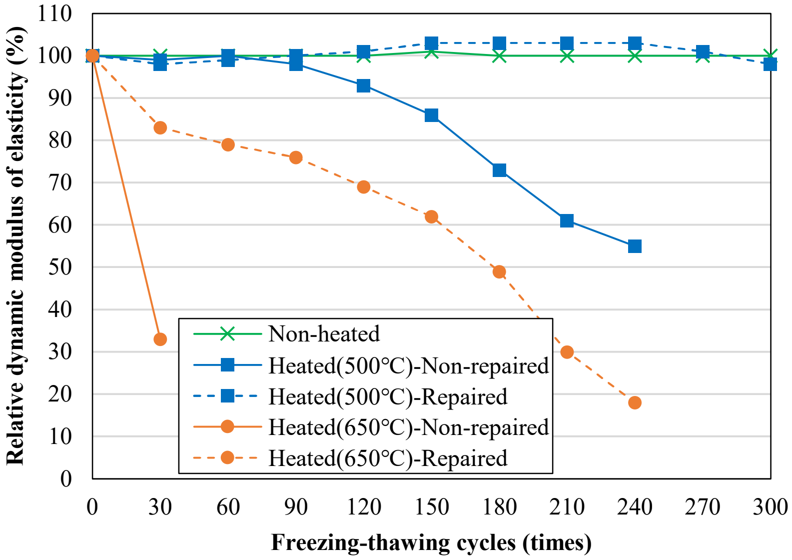


Fig. 5 Relationship between relative dynamic modulus of elasticity and freezing-thawing cycles.

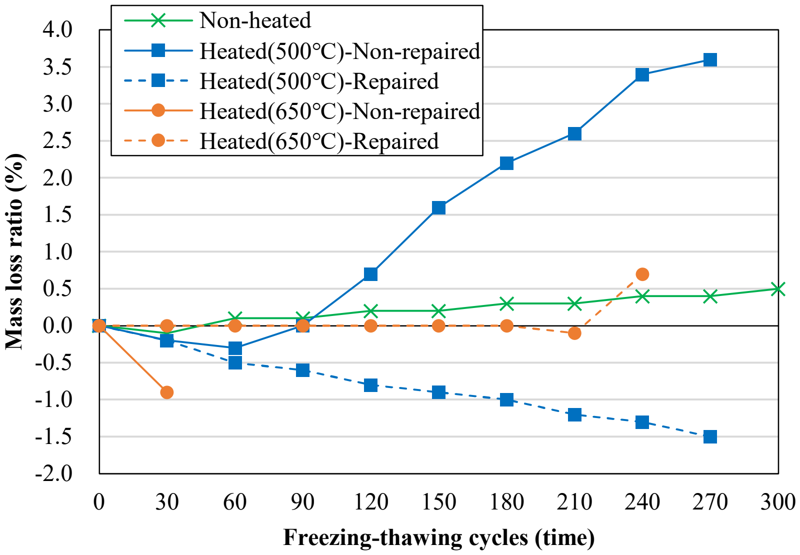
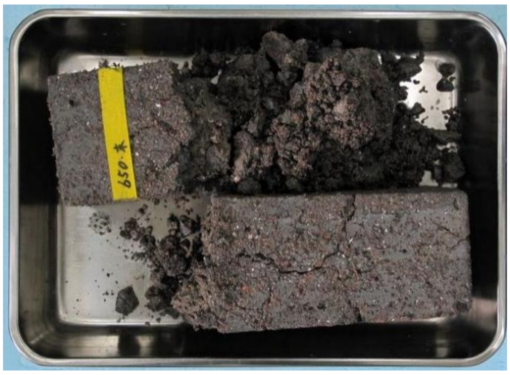


Fig. 6 Relationship between mass loss ratio and freezing-thawing cycles.

Table 6 Surface appearance of specimen before and after the freezing-thawing test.

(a-1) Heated (500°C)-non-repaired, 0 cycle	(b-1) Heated (500°C)-repaired, 0 cycle
	
(a-2) Heated (500°C)-non-repaired, 270 cycles	(b-2) Heated (500°C)-repaired, 270 cycles
	
(c-1) Heated (650°C)-non-repaired, 0 cycle	(d-1) Heated (650°C)-repaired, 0 cycle
	
(c-2) Heated (650°C)-non-repaired, 30 cycles	(d-2) Heated (650°C)-repaired, 240 cycles
	
(c-3) Heated (650°C)-non-repaired, 60 cycles	(d-3) Heated (650°C)-repaired, 270 cycles
	

fspecimen) and the surface layer (0-10 mm from the surface of the specimen). When we collected the samples, coarse aggregate was avoided, thus the samples were matrix mortar.

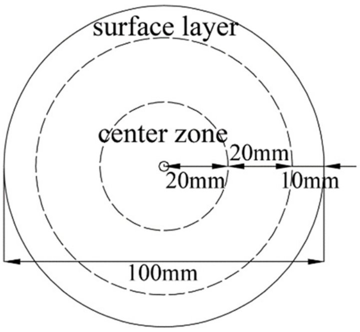


Fig. 7 Location of sample collection for XRD and SEM analysis.

The XRD analysis conditions were 40 kV and 30 mA of X-ray power, with a scanning speed of 2 degrees/min., a step size of 0.02 degrees, and a scanning range from 3-70 degrees (2θ). Cracks on a fractured surface of non-polished mortar samples may be produced at weaker locations during collecting from concrete, thus the fractured surface is not representative of the material. Especially, the reduced strength of heated concrete makes collecting easy to cause cracks. Therefore, in the SEM analysis, the polished samples were used. The mortar samples were dried and embedded in epoxy resin. After the epoxy resin hardened, the samples were polished while spraying water. No epoxy resin penetration into the inside of the samples was observed on the polished surfaces. In order to protect the pore structure from damage by polishing or to quantitatively evaluate the pore volume based on SEM images, the samples were impregnated by epoxy resin in vacuum in prior to embedding the epoxy resin (Kjellsen *et al.* 2003; Igarashi *et al.* 2003). However, vacuum epoxy resin impregnation is very difficult for the mortar sample collected from the unheated concrete of series C60 because of its dense structure. Therefore, in order to make the comparison under the same conditions, all matrix mortar samples were not epoxy resin-impregnated. SEM images were obtained at magnifications of 100 and 2500, and EDS analysis was performed on 4 or 5 randomly distributed points, avoiding aggregate particles as much as possible, and the contents of Si, Na, and Ca elements were measured.

4.2 Results of analysis and discussion

4.2.1 XRD results

XRD patterns of Series C24 samples are shown in **Fig. 8(a)**. In the non-heated sample, portlandite ($Ca(OH)_2$), calcite ($CaCO_3$), albite ($NaAlSi_3O_8$), orthoclase ($KAlSi_3O_8$), and quartz (SiO_2) were mainly detected. Carbonation of the portlandite occurred during storage and curing of the specimens. Albite, quartz, and orthoclase originated in aggregates. Calcite increased, but portlandite decreased in the heated/non-repaired sample. This is because during heating and after heating, portlandite easily reacts with CO_2 gas to form $CaCO_3$ (Atarashi *et al.* 2022) and the portlandite decomposed. Compared to the heated/non-repaired sample, the heated/repaired samples had fewer calcite, suggesting that the repair using the NW-LS solution decreased the carbonation reaction after heating, and thus the repaired concretes had high carbonation resistance.

The surface layer sample of the heated/repaired concrete contained more calcite, but fewer portlandite compared to the sample collected from the center, indicating that in the surface layer, there were more carbonation reactions before the repair, and more silicate reaction with LS after the repair. During cooling and storage of the heated concrete specimens, atmospheric CO_2 penetrated from the surface to the inside. Thus, there were more carbonate reactions in the surface layer. Although the NW-LS solution could penetrate into the center of the heated concrete specimens, the effective ingredient (Si^{4+} ions of lithium silicate) was more in the surface layer than in the center, as stated in our previous paper (Kitada and Li 2024) and as descried in the SEM-EDS analysis later. Therefore, the silicate reactions in the surface layer were estimated to be more than in the center.

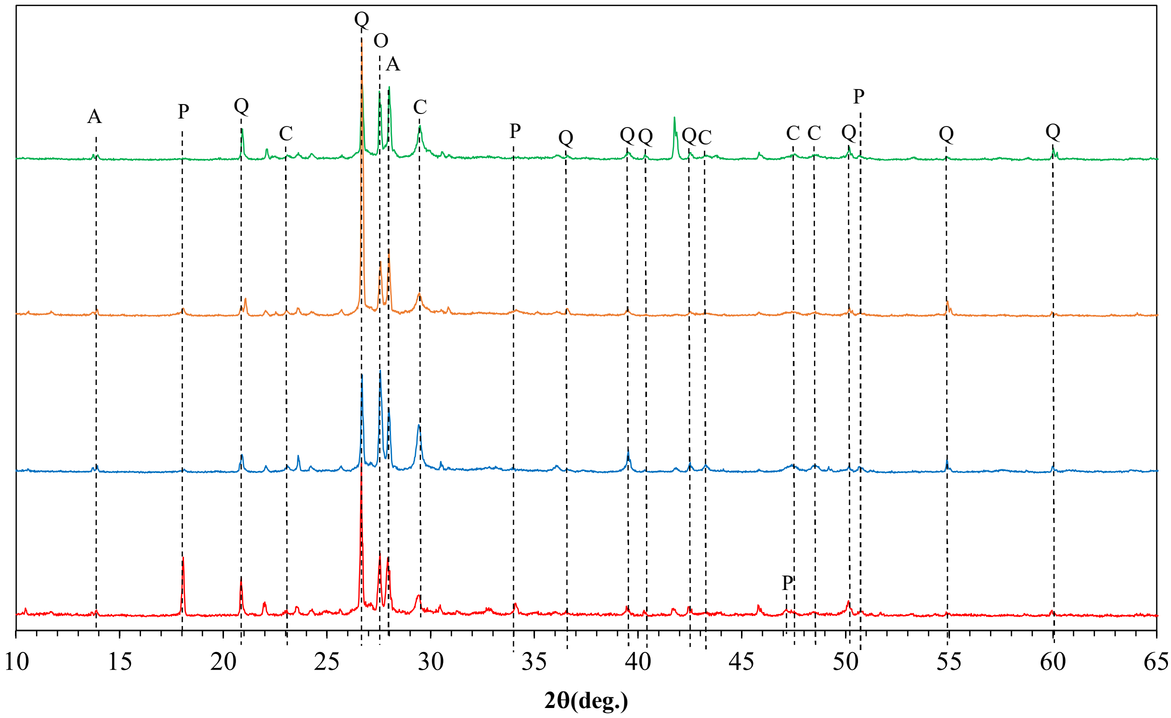
XRD patterns of the C60 samples are shown in **Fig. 8(b)**. Portlandite and calcite were present in all of the C60 samples, but the non-heated sample had more portlandite and calcite. The reason for the small amount of portlandite in the heated/non-repaired concrete is thought to be that part of $Ca(OH)_2$ decomposed during 650°C-heating. The reason why the portlandite in the two heated/repaired concrete samples collected from the surface layer and the center of specimen, respectively, was lower than that in the non-heated specimen is thought to be due to the reaction between the $Ca(OH)_2$ or CaO from the decomposed $Ca(OH)_2$ in the concrete and the silicate from the NW-LS solution. However, not only the heated/repaired concrete, but also the heated/non-repaired concrete had very little calcite. That is to say, high-strength concrete still has high carbonation resistance (small carbonation depth) after heating. This XRD analysis result is consistent with the result of the accelerated carbonation test, as shown in **Table 5** and **Fig. 4(b)**.

In the range 25-30° of all the eight XRD patterns in **Fig. 8**, halo peaks were observed, indicating the presence of C-S-H gels in the eight samples. Because the product of silicate reaction between LS and portlandite is C-S-H gel, we could not judge whether the silicate reaction occurred or not from the halo peaks. However, from the reduction of portlandite in the heated/repaired concrete compared to the non-heated concrete, and from the reduction or non-change of calcite in the heated/repaired concrete compared to the heated/non-repaired concrete, we can judge the occurrence of silicate reaction in the heated/repaired concretes. It is noted that in the XRD patterns of 650°C-heated concrete samples, CaO peak was not observed. This may be because the CaO generated by the decomposition of $Ca(OH)_2$ by heating at 650°C reverted to $Ca(OH)_2$ with moisture during the storage of concrete specimen after heating or during the preparation of XRD samples.

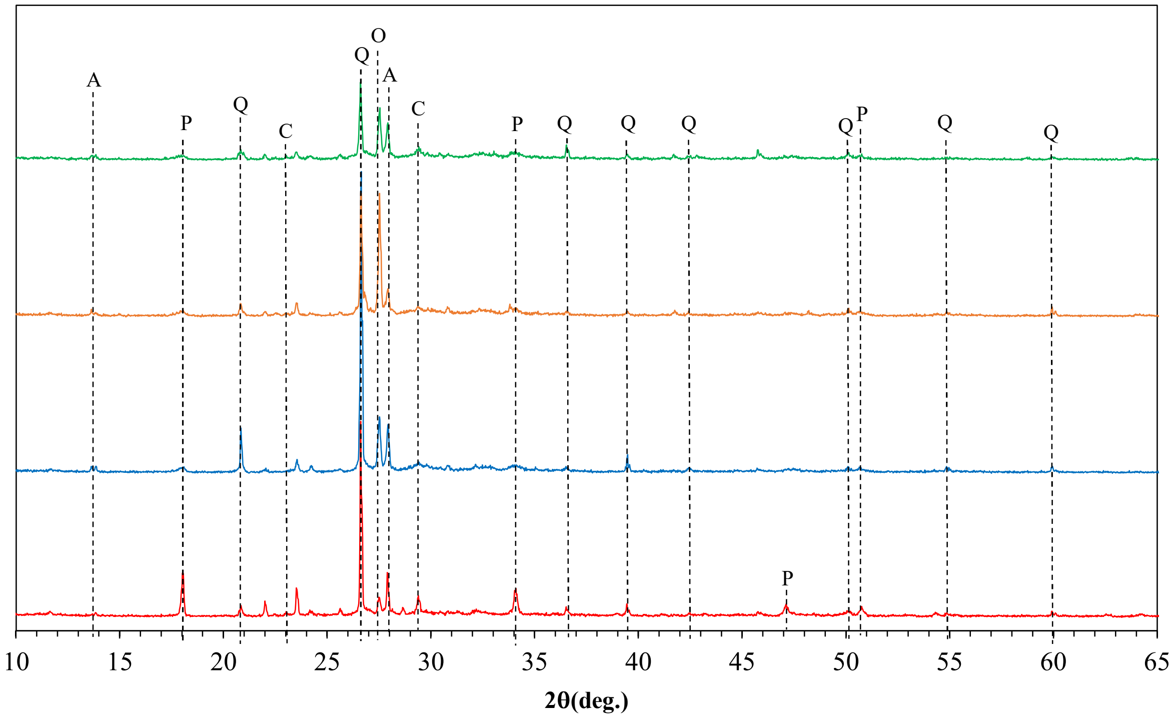
4.2.2 Results of SEM-EDS analysis

The SEM images of Series C24 and Series C60 are shown in **Figs. 9** and **10**, respectively. For each pattern, the left-hand side images with low magnification of 100 show the micro-structures of matrix mortars between coarse aggregates. However, microstructural changes caused by heating and repairing are barely visible in these low magnification SEM images. Therefore, the authors further observed the microstructure of the cement paste matrix between the fine aggregates at multiple locations on the 100-magnification images using high magnification of 2500. The right-hand side images with 2500-magnification image of each pattern shows the micro-structure of a randomly selected area of cement paste matrix, which is marked with a rectangle in the left-hand side 100-magnification image. Although the 2500-magnification image is given here for only one site on the 100-magnification image, the images of cement paste matrix at other sites present the same or similar characteristics. Because these 2500-magnification SEM images have the same magnification, the relative denseness between the samples can be determined by observing and qualitatively comparing these images.

As shown in the right-hand side images of **Figs. 9(a)** and **10(a)**, the non-heated/non-repaired samples exhibit fewer voids and discontinuities. However, the heated/non-repaired samples had many voids or/and fragments as seen in the right-hand side images of **Figs. 9(b)** and **10(b)**, indicating the dehydration of hydrates including $Ca(OH)_2$, and C-S-H, etc. (Peng and Huang 2008). The two samples for each series, which were heated and repaired with the NW-LS solution, have good structural integrity, with greatly reduced discontinuities, compared to the heated/non-repaired samples, which was attributed to the reaction between the NW-LS solution and the Ca^{2+} in concrete, and the rehydration of decomposed hydrates.



(a) Series C24

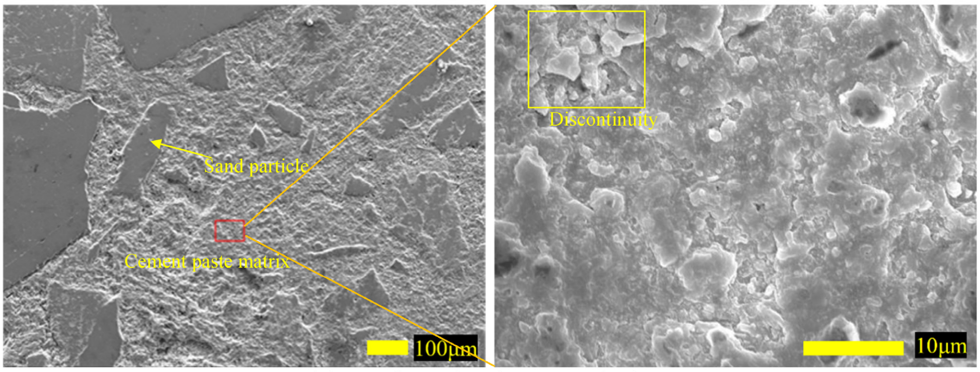


(b) Series C60

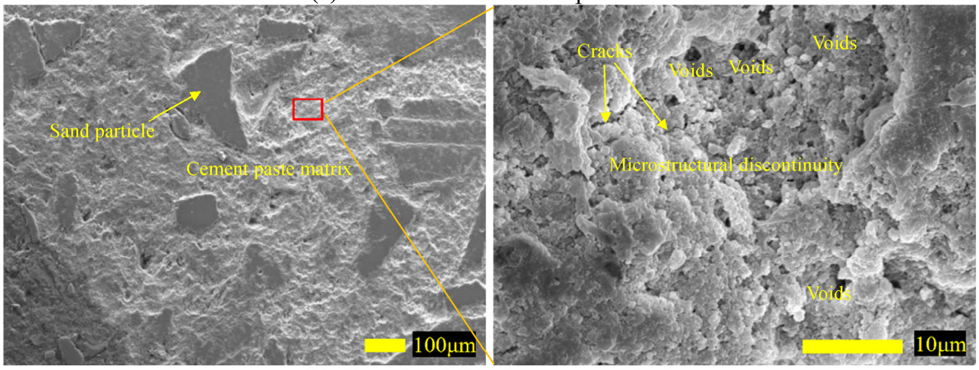
[Notes] A: albite, $NaAlSi_3O_8$; C: calcite, $CaCO_3$; O: Orthoclase, $KAlSi_3O_8$; P: portlandite, $Ca(OH)_2$; Q: quartz, SiO_2.

Fig. 8 XRD patterns of non-repaired and repaired concretes.

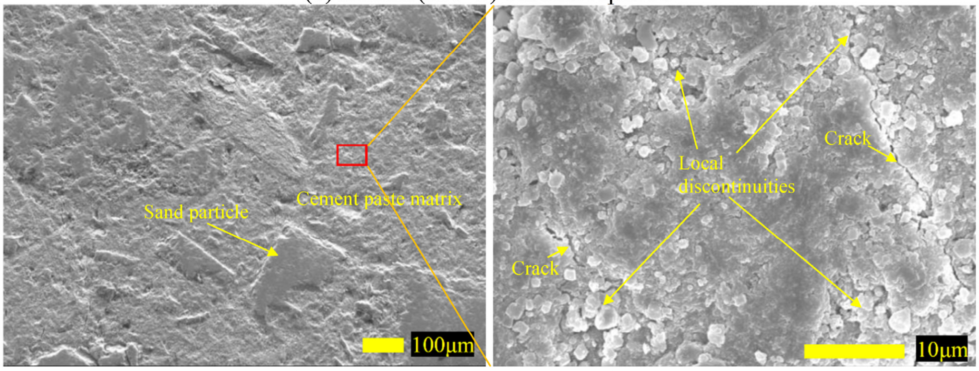
As stated earlier, the NW-LS solution can supply water for the rehydration. However, the heated/ repaired samples still had higher micro-structural discontinuities than the unheated samples. As shown in the right-hand side images of **Figs. 9(c)**, **9(d)**, **10(c)**, and **10(d)**, the heated/repaired samples had cracks, even the gel fragments, indicating they were not fully healed. Therefore, even if the mechanical performances of the



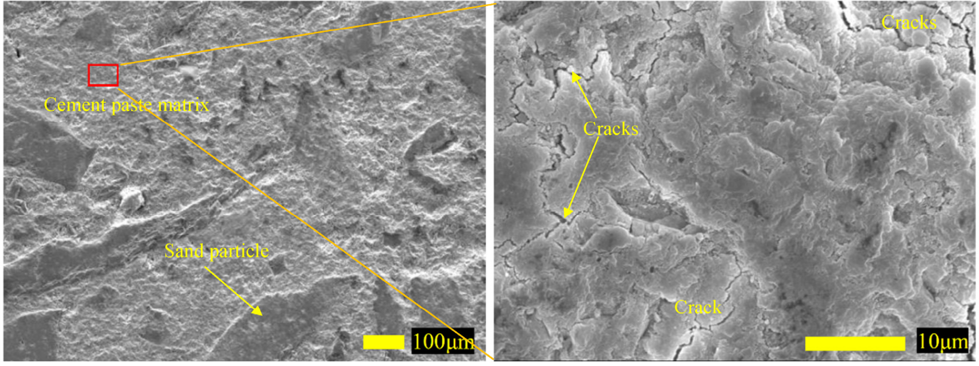
(a) Non-heated and non-repaired



(b) Heated (650°C) but non-repaired



(c) Heated (650°C) and repaired (Center zone)

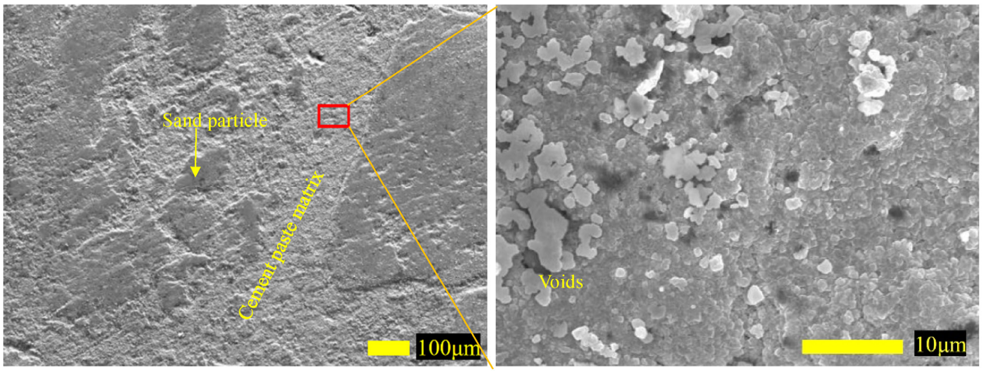


(d) Heated (650°C) and repaired (Surface layer)

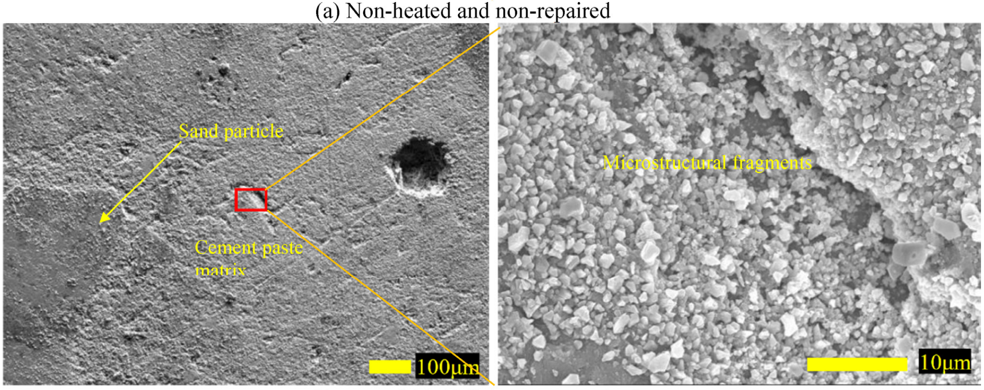
Fig. 9 SEM image of Series C24 (left: at 100-magnification, right: at 2500-magnification). The images on the right are enlargements of the marked areas in the images on the left.

specimens, which were heated at 650°C and repaired with the NW-LS solution, did not return to their original levels before heating.

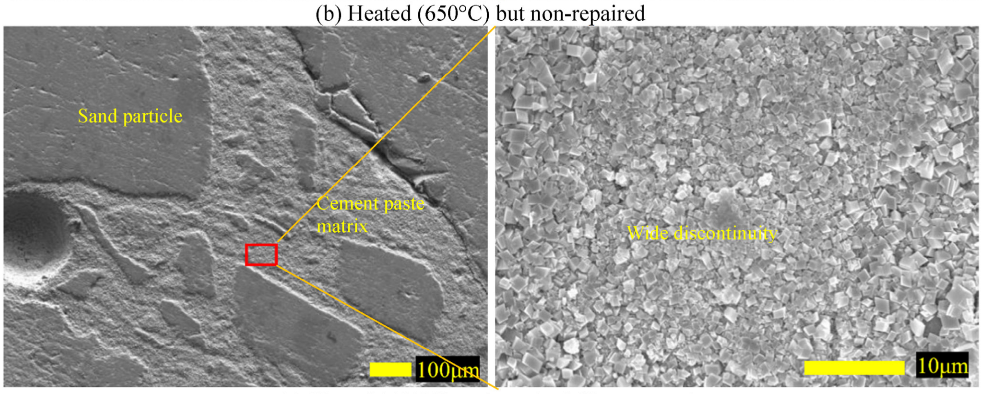
By comparing **Figs. 9(c)**, **9(d)**, **10(c)**, and **10(d)**, we found that the heated/repaired C24 samples had fewer voids and better micro-structural integrity than the heated/repaired C60 samples. This can explain why the mechanical properties of C24 concrete recovered at a



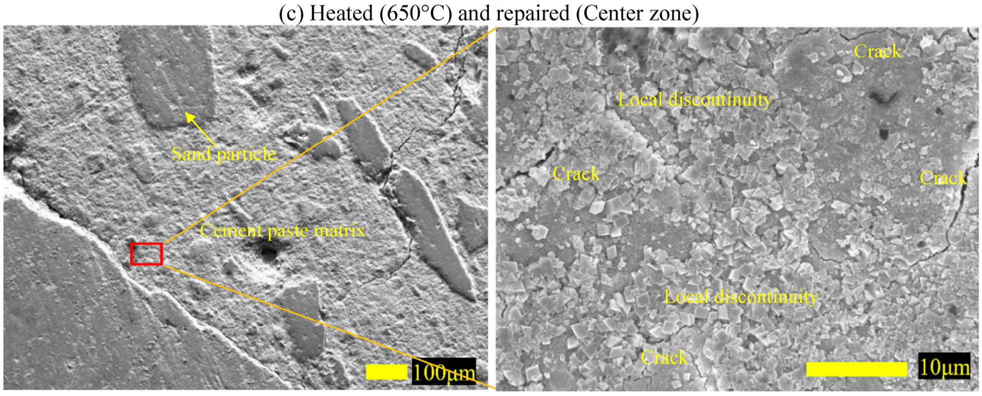
(a) Non-heated and non-repaired



(b) Heated (650°C) but non-repaired



(c) Heated (650°C) and repaired (Center zone)

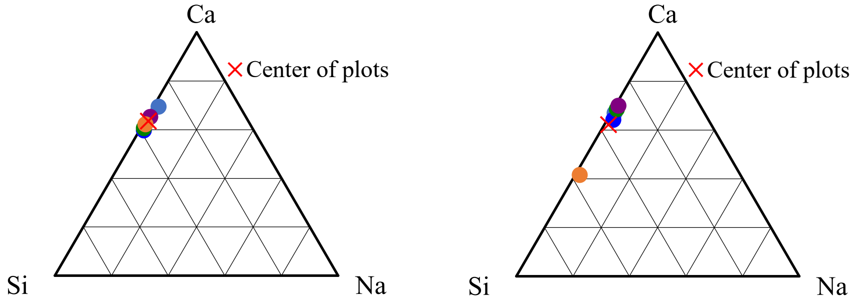


(d) Heated(650°C) and repaired (Surface layer)

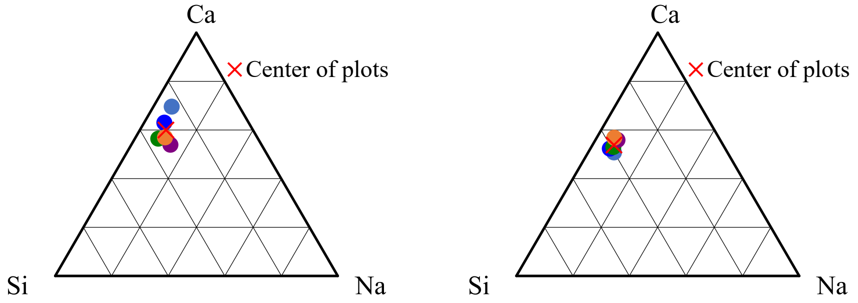
Fig. 10 SEM image of Series C60 (left: at 100-magnification, right: at 2500-magnification). The images on the right are enlargements of the marked areas in the images on the left.

higher rate than those of C60 concrete after heating at 650°C and then repairing.

The contents (mass%) of elements Si, Na, and Ca in the four samples of each series of concrete were plotted in the ternary diagrams of Si-Ca-Na (**Figs. 11** and **12**), which were obtained by the SEM-EDS analysis, in order to clarify the permeability of NW-LS solution and the reason for the differences in the degree of repairing between the center zone and the surface layer. The analyzed points were located in the areas of the 2500-

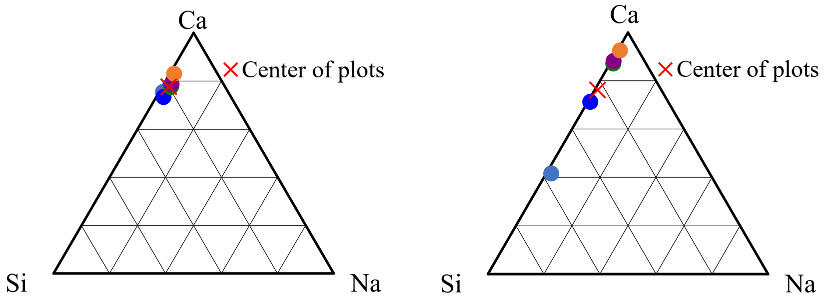


(a) Non-heated and non-repaired (b) Heated (650°C) but non-repaired

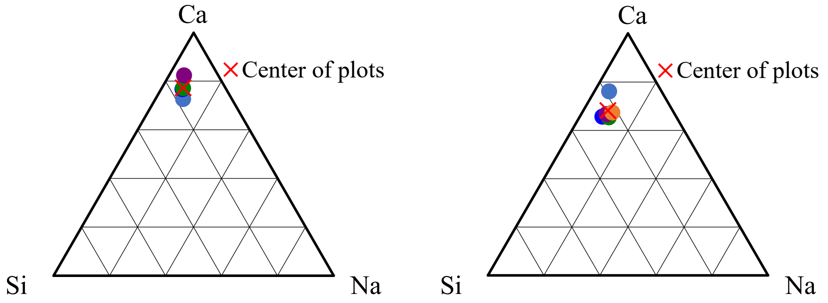


(c) Heated (650°C) and repaired (Center zone) (d) Heated (650°C) and repaired (Surface layer)

Fig. 11 Ternary diagrams of Si-Ca-Na for the samples of Series C2.



(a) Non-heated and non-repaired (b) Heated (650°C) but non-repaired



(c) Heated (650°C) and repaired (center zone) (d) Heated (650°C) and repaired (surface layer)

Fig.12 Ternary diagrams of Si-Ca-Na for the samples of Series C60.

magnification SEM images shown in **Figs. 9** and **10**. As shown in **Figs. 11** and **12**, the centers of the plot distributions of the two non-repaired samples of each series were almost identical, and the sodium (Na) content was close to zero. However, the sodium (Na) content increased in the two repaired samples of each series, and the plots shifted to the Si apices. This confirmed that the NW-LS solution penetrated into the centers of the cylindrical specimens. Moreover, the centers of the plots of elemental contents for the surface layers of the two repaired cylindrical specimens were closer to the Si apices, compared to the centers of the plots of elemental contents for the central zones of the two repaired cylindrical specimens. Thus, we can judge that the amount of the effective ingredient (lithium silicate) of the NW-LS solution penetrating into the center zone of the cylindrical specimen of each series was less than that in the surface layer. This explains why the center zones were not as dense as the surface layers in the repaired cylindrical specimens. We will conduct further investigations by increasing the concentration of lithium silicate in the NW-LS solution.

5. Summary

In this study, the highly permeable and alkaline silicate-based solution with a Na/Si molar ratio of 1.25 and a solid concentration of 16.6% (called NW-LS solution here), which consists of sodium hydroxide (NaOH), lithium silicate (LS) and water, was used to repair fire-damaged concrete. We investigated in detail the degree of recovery of compressive strength, carbonation resistance and freezing-thawing resistance of concretes. The concretes had different strength grades (20-60 MPa) before heating, and were heated at different temperatures (300, 500, 650°C) and cooled in water or in air, and then immersed in the NW-LS solution for repairing. Moreover, XRD and SEM-EDS analyses were also conducted to investigate the changes in compositions and internal structure of heated concrete after the repairing. The obtained results are summarized below.

1) The residual compressive strength of the concretes after heating and cooling in air, which had compressive strengths of 24-60 MPa before heating, decreased to 80-88% at a heating temperature of 300°C, 37-57% at 500°C, and 30-42% at 650°C.
2) After repair with the NW-LS solution by the immersion method, the compressive strength of the concrete with the original strength of 24.4 MPa increased up to 113.6% at 300°C, 111.3% at 500°C, and 99.0% at 650°C. For the original strength of 42.7 MPa, the strength recovered to 88.9% at 300°C, 70.2% at 500°C, and 73.2% at 650°C. For the original strength of 60.0 MPa, the strength recovered to 87.4% at 300°C, 83.2% at 500°C, and 75.5% at 650°C. It can be said that normal strength concrete can be restored completely in respect of strength if heated below 650°C. Considering experimental errors of the 500°C-heated concrete with strength 42.7 MPa before heating, about 90%, 80%, and 70% of original strength of high strength concrete of 36 MPa or more can be recovered when heated at 300, 500, and 650°C, respectively. And the recovery rate of strength of air-cooled concrete after being repaired was higher that of water-cooled concrete.
3) The concretes heated at 500°C or 650°C exhibited significantly lower carbonation resistance and freezing-thawing resistance. Also, the lower the compressive strength before heating, the larger the decline of carbonation resistance after heating. However, the carbonation resistance and the freezing-thawing resistance can be significantly improved by repairing with the NW-LS solution. In particular, when the heating temperature was 500°C, the NW-LS solution was highly effective in restoring carbonation resistance and frost resistance of heated concrete. The NaOH in the NW-LS solution can increase the alkalinity of the heated concrete that may be partially carbonated during heating and after heating.
4) The internal structure after heating becomes loose due to the decomposition of hydrates, but can be refined by repairing with the NW-LS solution based on the SEM images. The NW-LS solution components were detected in the center of the heated and repaired specimen by SEM-EDS analysis, regardless of strength grade, confirming that the solution has a high permeability. However, there were fewer components in the center than in the surface layer.
5) XRD analysis confirmed that the heated/repaired concrete had less portlandite than the unheated concrete, less calcite than the heated/non-repaired concrete, and that the silicate reaction between LS and Ca^{2+} occurred in the heated/repaired concrete.

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