- Technical Paper -

EVALUATION OF RE-CURING FOR THE RECOVERY OF HIGH-STRENGTH MORTAR EXPOSED TO FIRE

Michael HENRY^{*1}, Tae-Ho AHN^{*2}, Yoshitaka KATO^{*3} and Toshiharu KISHI^{*4}

ABSTRACT

Re-curing of fire-damaged concrete can restore structural performance without removal and casting operations, reducing waste generation and resource consumption. In this research, the effect of water re-curing on the recovery of damage due to fire was investigated. Recovery of pore structure and crack self-healing were observed and attributed to both the rehydration of dehydrated cement as well as the hydration of unhydrated cement particles. Finally, in order to bridge the gap between mortar and concrete, damage and self-healing around aggregates was discussed.

Keywords: high-strength concrete, fire damage, repair, crack self-healing, mechanical recovery

1. INTRODUCTION

Concrete exposed to high temperatures undergoes a reduction in performance, such as decreased load-carrying capacity and durability. In order to restore these properties, repair operations should be carried out, which typically involves the removal of damaged areas and the casting of a patching material [1]. Past research has found that re-curing fire-damaged concrete in water after heat exposure can restore strength and durability [2]. Therefore, a repair method utilizing re-curing could potentially replace traditional repair operations in some cases, thus reducing waste generation and resource consumption.

For re-curing to be realized as a repair method, the mechanism by which re-curing can restore fire damage should be understood. However, the damage and re-curing mechanisms are not independent and the influence of the damage factors is not entirely clear. In a previous research on high-strength mortar damaged by fire, specimens' strength loss and recovery were attributed to changes in porosity and cracking caused by different cooling and re-curing conditions [3].

The progress of dehydration of the cement paste, which depends on the temperature gradient, has a direct bearing on the re-curing behavior (Fig. 1). The dehydrated zone is a brittle layer composed of sintered material which cannot undergo re-curing. The dehydrating zone is the region in which evaporation of free water and chemically-bound water is occurring, but the material has not yet been sintered. The quasi-saturated zone contains water under heating. Re-curing is most likely to be effective in the dehydrating zone, where supply of water can both rehydrate dehydrated cement, as well as hydrate unhydrated cement [5]. In the quasi-saturated and initial state zones, supply of water would serve primarily to hydrate the unhydrated cement grains which are found in high strength, low water-cement (W/C) ratio concretes. By supplying water and initiating the hydration reaction, hydration products fill the pore space and reduce porosity. Since porosity is a primary factor for determining strength and durability, a reduction in porosity should correspond with an increase in these properties.

In this research, the effect of water re-curing on porosity recovery and crack self-healing was investigated. In addition, the progression of dehydration under heating was discussed with relation to re-curing. While not explicitly observed here, the mechanism for cracking and self-healing around aggregates in concrete was proposed and the crack self-healing behavior of non-fired concrete was shown. By bringing together these different mechanical damage mechanisms and their respective recovery behaviors, a comprehensive view of the potential of re-curing for the recovery of fire-damaged concrete could be constructed.





^{*1} Doctoral student, Department of Civil Engineering, University of Tokyo, JCI Member

- *2 Project research associate, Institute of Industrial Science, University of Tokyo, JCI Member
- *3 Associate professor, Institute of Industrial Science, University of Tokyo, JCI Member
- *4 Associate professor, Institute of Industrial Science, University of Tokyo, JCI Member

2. TEST PROGRAMS

2.1 Materials

High-strength cement mortar was prepared from water, ordinary Portland cement (Type 1), Fujigawa river sand, and air-entraining and high-range water reducing admixtures. A water-cement ratio of 0.3 was used with a target 28-day compressive strength of 100 MPa. Complete mix proportions are given in Table 1.

2.2 Specimens

Cylinder specimens $(50 \times 10 \text{ cm})$ were cast using high-strength mortar. After casting, specimens were sealed and cured in the molds for 24 hours. They were then removed and placed in water curing (20°C) for 13 days, then transferred to air curing (20°C, 60% RH) until fire exposure at 28 days from casting.

2.3 Fire exposure

Fire exposure was emulated using an electric furnace. As this furnace does not have a control mechanism for the rate of heat increase, the furnace was preheated to the target temperature before beginning exposure. The temperature was set at 550°C for this experimental program. This value was chosen by performing a trial series of tests in order to determine, for this specimen shape, size and mix proportions, the temperature at which the amount of fire damage was the highest yet the probability of spalling the lowest. A 2-hour exposure time was also selected based on a trial experimental series. This exposure condition was assumed to yield complete dehydration at the surface, with a profile similar to Figure 1.

2.4 Re-curing conditions

After removal from heating, specimens were placed in one of two re-curing conditions. Water-cooled and re-cured specimens were placed into room-temperature (20°C) water immediately after removal from heating; air-water (AW) re-cured specimens were allowed to cool in the air for one hour before water submersion. Specimens used for investigating the self-healing of cracks were allowed to cool in the air until surface cracking was observed, and then re-cured in water.

2.5 Specimen testing

Testing consisted of three phases. The first phase measured strength and porosity recovery; the second phase observed cracking patterns using epoxy injection; the third phase observed crack self-healing behavior by microscope photography.

Compressive strength was measured under the unstressed residual condition. Values reported are the average of three tests. Mercury Intrusion Porosimetry (MIP) specimens, approximately 5 millimeters in size, were taken from compressive strength specimens (upper half, outer 1.5 centimeters) after testing to maintain consistency between tests, submerged in acetone for 24 hours to stop the hydration reaction, and then dried using a D-dry vacuum pump. Compressive strength and MIP specimens were tested before heating, 1 hour after heating, and after 3 and 28 days.

In order to observe the cracking pattern and crack recovery under different re-curing conditions, an epoxy injection procedure was applied [6]. After re-curing, epoxy was injected via vacuum pressure. This process can inject epoxy into cracks as small as 6 micrometers, and the hardening of the epoxy helps to prevent the formation of secondary defects. After epoxy injection, specimens were cut in section 4 centimeters from the top of the specimen, and then placed under ultraviolet (black) light. The dispersion of epoxy was then recorded by photograph. Water re-cured (1 hour and 7 days) specimens were measured.

Microscope observation was performed to investigate the progress and mechanism of crack self-healing. Specimens in this test series were allowed to cool in the air until surface cracking occurred, then placed in water to initiate self-healing. Observation was performed after 1, 3, 7, and 28 days.

3. RESULTS

3.1 Strength and porosity comparison

Strength and porosity results are shown in Figure 2 for water and air-water re-cured specimens. By 28 days, water and air-water specimens recovered to 72.4% and 84.5% of their pre-fired strength, respectively. Water specimens underwent a strength loss of 61.5% but recovered 33.9%; air-water specimens lost 29.5% and recovered 14.0%. The effect of thermal shock on strength can be observed in the 33.4% difference in strength 1 hour after the fire.

Table 1 Mortar Mix Proportions

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Cement (kg/m ³)	С	230
Water (kg/m ³)	W	767
Fine aggregate (kg/m ³)	S	1380
Air entraining agent (%C by weight)	AE	0.4
High-range water-reducing admixture (%C by weight)	HRWA	1.5
Water/cement ratio	W/C	0.3
Sand/cement ratio	S/C	18



Porosity loss and recovery can also be observed for both re-curing conditions. After heating, porosity increased by 67.1% and 72.7% for water and air-water, respectively. Water specimens recovered 65.4% for a final porosity 1% larger than the original value. Air-water specimens recovered only 55.0% to 17.6% larger than the original porosity. There was no significant thermal shock effect on the porosity, as both series of specimens exhibited nearly the same porosity after 1 hour of cooling.

3.2 Pore structure re-curing behavior

In order to observe the porosity behavior in greater detail, the pore distributions before heating, after heating, and after 28 days re-curing are shown in Figure 3 for water specimens and Figure 4 for air-water specimens. 3-day results were omitted for clarity.

Heating caused a significant increase in the average pore size for both water and air-water specimens. Before heating, the pore fraction was concentrated between 3 and 40 nanometers; after heating, between 20 and 1000 nm, with a much larger occupied volume. In both cases, re-curing led to the recovery of the pore structure by 28 days.

A comparison of the 28-day re-cured pore structures for water and air-water specimens is given in Figure 5. For both water and air-water, re-curing after fire resulted in a greater volume of smaller pores than before heating. However, pores in the water specimens occupy less volume than in air-water specimens. Measurement of the weight of water specimens (1 hour after water submersion) and air-water specimens (1 hour air, 1 hour water) found that water specimens absorbed 44.7 grams of water compared to the 37.5 grams for air-water specimens.

3.3 Cracking observation by epoxy injection

Cracking of water specimens observed utilizing epoxy injection can be seen in Figure 6. Light areas in the image show where epoxy penetrated through cracks; dark areas are un-cracked, or where epoxy didn't penetrate.

The effect of immediate water submersion is shown in the left figure. The epoxy can be seen in small, spidery radial lines, which indicates the occurrence of micro-cracking. Visual inspection of these specimens showed no visible defects. Almost no epoxy is visible in the right figure, which shows that epoxy was unable to penetrate due to the self-healing of the micro-cracks under water supply.

3.4 Crack self-healing microscopic observation

Microscopic observation of crack self-healing under water supply is shown in Figure 7 for 1, 3, 7, and 28 days of water re-curing. Specimens in this case were allowed to air cool until surface cracks formed, and then submerged in water. The initial crack width, marked in the top image, was 0.31 millimeters.

Water re-curing was effective in causing the self-healing of the crack. Significant regrowth could be observed by 7 days, and almost complete self-healing was seen by 28 days.





Fig.4 Pore distribution for air-water re-curing



Fig.5 Comparison of re-cured pore distributions after 28 days re-curing

4. DISCUSSION

4.1 Porosity and pore structure

The recovery of porosity and the pore structure was shown in Figures 3, 4, and 5. It was shown that, after 28 days re-curing, the distribution of pores for both water and air-water specimens were different than before heating; pores tended to be of a smaller diameter but occupied a greater volume. In order to understand this phenomenon, the source of pore structure recovery should be discussed. It is believed that CaO, produced from the decomposition of CH and $CaCO_3$, as well as calcium silicate compounds, from the decomposition of C-S-H by heating, can be rehydrated by the supply of water after heating exposure [7]. If this were the only source of recovery, the recovered pore structure should be similar in nature to the original. However, as can be seen in the previous figures, the concentration of pore diameters after re-curing is less than before, so some other reaction has taken place to reduce the pore sizes further. Typically, this is attributed to the hydration of unhydrated cement grains in the concrete matrix, and has been mentioned in previous research works.

The significant increase in pore sizes due to heating may allow for the supply of water to unhydrated cement which was previously blocked by the dense pore structure typical to low W/C concrete. After heating, the pore size is significantly larger and occupies a larger volume, thus allowing the transport of water quickly into the pore structure and initiating the hydration of unhydrated cement grains as well as the rehydration of dehydrated products. While the relative influence of unhydrated and rehydrated products on the recovery of pore structure is not clear, it would be expected that, in older structures or structures under constant water supply (like tunnels or underwater structures), the effect of unhydrated cement would decrease.

4.2 Cracking and crack self-healing

The strength reduction observed in Figure 2 can be attributed to three factors: porosity (discussed in the previous section), chemical deterioration (not investigated in this research), and cracking. While the relative influence of each of these factors on the strength recovery is not clear, self-healing of cracks was observed under water supply (Figures 6 and 7).

Self-healing behavior in plain concrete, for low W/C ratios, is typically attributed to the hydration of unhydrated cement particles [8]; therefore, self-healing after fire damage could be attributed to the same mechanism. However, in the case of fire damage, rehydration of dehydrated calcium silicate compounds may also contribute to crack recovery.

When comparing the relative recovery of strength, porosity, and cracking self-healing, it can be seen that, even though porosity recovers to near pre-fired levels and cracks achieve significant self-healing, at 28 days the strength has still not completely recovered. Incomplete strength recovery may possibly be due to the structure of the product which fills the crack after self-healing. At 28 days, while hydration and rehydration product can be seen to almost completely fill the crack (Figure 7, bottom), there is some discoloration in the area of the new product, as well as some small regions which are not completely filled. If the crack size is significantly large enough, then self-healing may not occur no matter the length of time. Furthermore, the mechanical strength of the new product filling the crack is not entirely known; these may play a factor in preventing complete strength recovery.



Fig.6 Cracking of water specimen 1 hour after heating (left) and 7 days water re-curing (right)



Fig.7 Crack self-healing at (from top) 1 day, 3 days, 7 days, and 28 days

4.3 Self-healing around aggregates

Another factor which may prevent complete strength recovery is the self-healing which occurs around aggregates in concrete. While not explicitly studied in this research, the concept will be proposed for future consideration.

Under sufficiently high heating conditions, cement paste begins to shrink due to the evaporation of free and chemically bound water. However, some types of aggregate, such as siliceous, undergo an increase in volume due to crystal transformations caused by the increase in temperature. This creates stresses in the region around the aggregate, which may be released in the form of cracks. For re-curing to be utilized as an effective recovery method, the re-curing behavior around these types of cracks should be considered.

Cracking around an aggregate and the self-healing behavior in this area is shown in Figure 8 for non-fired concrete. In the region where both faces of the crack are cement paste, the crack self-healing occurs from both sides, so complete recovery can occur. However, where one side of the crack is aggregate, only partial self-healing behavior was observed. Therefore, while this behavior has not been studied or observed in fire-damaged concrete explicitly, it can be assumed that self-healing around aggregates will occur similar to that of non-fired concrete. As a result, this type of cracking requires special consideration for realizing the potential of re-curing as a recovery method.

4.4 Secondary spalling due to re-curing

Re-curing, which generally restores strength and durability by reducing porosity and healing cracks, may also cause damage due to the expansion caused by hydration and rehydration. Although mentioned in previous research [9], this concept was not directly related to the concept of dehydration zones. The mechanism is most easily viewed at the surface and around aggregates, where the bond strength after heating is weak. When considering the dehydration zones, shown again in Figure 9, the dehydrated zone can be characterized as a thin, brittle layer of sintered material with no capability for re-curing. However, the dehydrating zone below that can undergo re-curing through hydration and rehydration of unhydrated cement and dehydrated cement, respectively. During re-curing of the dehydrating zone, expansion will occur due to the hydration/rehydration reactions. This will place stresses on the dehydrated zone and may result in a kind of secondary spalling, in which the expansive force breaks the weak bonds of the outer layer and causes the surface layer to break away. While the full extent of this effect is uncertain, it may result in merely an unsightly appearance if the outer layer is thin, or could potentially have a greater effect on mechanical sense or durability if the spalling is deeper.

Aggregates may also cause secondary spalling. As explained in the previous section, cracking occurs around aggregates due to stresses caused by incompatible thermal deformation with the cement paste. If the aggregate is located near the surface, then expansion due to re-curing in the dehydrating zone may cause aggregate pop-out due to the weakened bond between the aggregate and the surrounding paste caused by cracking.

4.5 Proposal of a chemical bonding agent

In the previous section, it was proposed that expansion due to re-curing may destabilize the dehydrated, sintered layer of material. Stresses caused by rehydration of the inner layers may exceed the strength of the brittle bonds in the sintered zone and result in secondary spalling at the surface, either as cement flaking or aggregate pop-out. A chemical bonding agent, which could increase bond strength and prevent the occurrence of secondary spalling, would increase the feasibility of re-curing. Such an agent could be delivered as some fraction part of the re-curing water and, since water is transported from the outer face inwards, the agent would be able to strengthen the outer, dehydrated zone and aggregate interfaces before hydration and rehydration expansion could destabilize and cause damage.



Fig.8 Example of partial self-healing around aggregate in non-fired concrete [10]



Fig.9 Damage due to expansion of dehydrating zone under water supply

5. CONCLUSIONS

- (1) In order to realize re-curing as a repair method for fire-damaged concrete, the mechanism for loss and recovery of mechanical sense and durability needs to be fully understood. In this research, high-strength mortar was utilized for experimentation; however, in order to bridge the gap between mortar and concrete, which is typically used in the field, damage and recovery concepts were proposed in addition to the observed behavior.
- (2) Strength loss was affected by an increase in porosity, caused by damage to the pore structure and shrinkage due to the evaporation of free and chemically-bound water, and cracking, caused by differential shrinkage in the specimens. Thermal shock, due to immediate water submersion, significantly reduced strength, but had little observable effect on the porosity, indicating the influence of the observed micro-cracks on strength reduction.
- (3) Strength recovery was driven by both the hydration of unhydrated cement and the rehydration of dehydrated cement products. These two chemical reactions lead to a decrease in porosity and the filling of pore spaces, as well as crack self-healing due to expansion during hydration and rehydration reactions and crystalline regrowth.
- (4) The damage mechanism and self-healing of cracks around aggregates in concrete was conceptually proposed. An example of partial self-healing in non-fired concrete was given to demonstrate the type of behavior that could be expected during re-curing.
- (5) While re-curing is intended to restore the performance of concrete, expansion under re-curing may destabilize the sintered outer layers, resulting in secondary spalling either as cement flaking or aggregate pop-out. The effect of this damage needs to be studied in order to understand the scale of this damage. One solution may be the development of a chemical bonding agent, which could act to restore bonding in the brittle layer and between aggregates and cement paste.
- (6) The relative influence of hydration and rehydration was found to be an important factor in understanding the driving mechanism for porosity recovery and crack self-healing. Analyses focusing on the changes in chemical composition at different stages of exposure and re-curing are recommended to clarify these factors.

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