Effect of Addition Rate of Expansive Additive on Autogenous Shrinkage and Delayed Expansion of Ultra-High Strength Mortar

Yulu Zhang, Atushi Teramoto, Taka-Aki Ohkubo

Abstract—In this study, the effect of expansive additives on autogenous shrinkage and delayed expansion of ultra-high strength mortar was explored. The specimens made for the study were composed of ultra-high strength mortar, which was mixed with ettringite-lime composite type expansive additive. Two series of experiments were conducted with the specimens. The experimental results confirmed that the autogenous shrinkage of specimens was effectively decreased by increasing the proportion of the expansive additive. On the other hand, for the specimens, which had 7% expansive additive, and were cured for seven days at a constant temperature of 20°C, and then cured for a long time in either in an underwater, moist (Relative humidity: 100%) or dry air (Relative humidity: 60%) environment, excessively large expansion strain occurred. Specifically, typical turtle shell-like swelling expansion cracks were confirmed in the specimens that underwent long-term curing in an underwater and moist environment. According to the result of hydration analysis, the formation of expansive substances, calcium hydroxide and alumina, ferric oxide, tri-sulfate contribute to the occurrence of delayed expansion.

Keywords—Ultra-high strength mortar, expansive additive, autogenous shrinkage, delayed expansion.

I. INTRODUCTION

ULTRA-HIGH strength concrete (UHSC) has high compressive strength and a dense inner structure, which suppresses the ingress of damaging factors thereby improving the durability of the building structure. However, the occurrence of shrinkage cracks at the age when the concrete is not sufficiently hardened has become a problem for UHSC. For concrete with a low water to binder ratio (W/B), the main cause of shrinkage cracks occurring early in the hardening process is the autogenous shrinkage of cement paste. Since this shrinkage occurs before the tensile strength of the concrete is developed, it is difficult to implement crack control measures after construction of the building. Consequently, expansive additives or shrinkage reduction agents are considered as a countermeasure for suppressing shrinkage cracks during the designing mix proportion stage.

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In order to establish the optimum usage of expansive additive for autogenous shrinkage reduction, our research group has conducted an experimental study on the autogenous shrinkage reduction mechanism of an expansive admixture. We used a cement paste composed of expansive additive and shrinkage reducing agent mixed into a silica fume premixed cement (SFPC) and having a water to binding agent ratio (W/B) of 16.5% [1], [2]. In conventional literature [3], it is said that the expandable hydration product, calcium hydroxide (CH) and ettringite (AFt) form, and impart an expansive effect on concrete, which reduces shrinkage cracks during the curing of concrete. In our group's previous research, to reduce the autogenous shrinkage of the ultra-high strength cement paste, two types of additive were tested. It has been confirmed that both ettringite-lime composite type and lime type expansive additive are effective for reducing the shrinkage in the early age. In addition, it becomes clear that after 24 hours, the formation of CH contributes to the autogenous shrinkage reduction effect of the expansive additive, which does not correlate with the formation of AFt. Furthermore, it was inferred that for the low W/B ratio cement paste, the autogenous shrinkage reduction effect due to formation of CH has no relationship with the category of expansive additive.

Since the production amount of CH has a correlation with the quantity of expansive additive it is possible to obtain substantial autogenous shrinkage reduction easily by adding more expansive additive. However, cases in which pop-outs and turtle shell-like swelling cracks occur due to delayed expansion of the expansive additive in the long term have been reported, when an excess of expansive additive is used.

In past investigations, deterioration cases of concrete due to delayed expansion of expansive additive were confirmed for both ettringite-lime composite type and lime type expansive additive. In the study by Suzuki et al. [4], the specimens, which have a W/B ratio of 15%, unit expansive additive quantity of 35 kg/m^3, and were cured over 100 days experienced delayed expansion. In the study of Jinnai et al. [5], the occurrence of delayed expansion was observed in specimens, which have a W/B ratio of 15%, unit expansive additive quantity of 40kg/m^3, and were cured in air after seven days of sealed curing. In the study of Guo et al. [6], it was confirmed that delayed expansion occurred in the specimens that had been cured for a long time in air after 7 days of sealed curing, and that had a W/B of 16.5% and an expansive additive quantity of 40kg/m^3. Considering the mechanism of delayed expansion prompted by an expansive additive, Guo described it as follows. In concrete with an

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extremely low W/B ratio and an excessive quantity of expansive additive, the unreacted expansive additive remains while hardening. With long term curing, the remaining expansive additive is hydrated with water supplied slowly from the aggregate, creating excessive expansion pressure, which results in the expansion deterioration of the concrete. Nevertheless, in the above research, which hydrate product contributes to the internal expansion pressure specifically, is unclear.

Delayed expansion of expansive additives when used excessively is mentioned in a similar expansion phenomenon that is caused by delayed ettringite formation (DEF). For concrete with high temperature curing at an early age, after several months to several years, the sulfate reacts with aluminate hydrate and generates AFt causing expansion and deterioration of the concrete [7]. It is known that AFt is the main cause of expansion pressure for DEF.

According to the previous studies [8]-[10], the conditions for the occurrence of DEF expansion are as follows: 1) high-temperature steam curing at the early age, 2) sufficient sulfate supply, in addition to 3) sufficient water supply. On the other hand, for UHSC, the delayed expansion due to expansive additives also occurs at the condition of: 1) no initial high-temperature curing, 2) extremely low W/B ratio, and 3) no external water supply. Although these conditions are clearly different for the two types of expansion, in either of them the concrete is degraded by excessive expansion pressure due to the formation of expansion hydrate after hardening. Additionally, since expansive additives contain a certain quantity of sulfate, it is possible that delayed expansion due to expansive additives occur under the same conditions as DEF expansion.

From these points of view, when using expansive additives, determining the shrinkage reduction mechanism and clarifying the occurrence condition of delayed expansion is required in order to establish a reasonable proportion of expansive additive that does not cause cracks and deterioration at either the initial or the long-term curing of UHSC.

This investigation was conducted to confirm the autogenous shrinkage reduction effect of expansive additives during early age, and clarify the conditions resulting in harmful delayed expansion due to expansive additive usage in UHSC.

II. OUTLINES OF EXPERIMENT

In this experiment, a number of ultra-high strength mortar specimens were prepared with different proportions of ettringite-lime composite expansive additive. Two series of experiments were conducted, series 1 focus on the autogenous shrinkage test before seven days, and series 2 concentrate to clarify the curing condition, which delayed expansion is occurred at after seven days.

A. Materials Used and Mixture Proportion of Mortar

Table I lists the materials used in this study. Table II lists the details of the mortar mixtures. All specimens had the same W/B ratio of 16.5% and the proportions of expansive additive in terms of the mass of the binding agent were 3%, 4%, 5%, and 7%. Fine aggregate of 30% by total volume was mixed into all

specimens, which was reported has induced to cause cracks due to delayed expansion [11]. To ensure the sufficient fluidity of casting, 3.1% superplasticizer (SP) by binding agent mass was added to all specimens. The 30% of the SP's mass was treated as solid content and 70% was treated as water when calculated.

TABLE I
PROPERTIES OF MATERIALS

Materials	Symbol	Properties
Cement	SFPC	Silica-fume pre-mixture cement, Density:3.04g/cm^3 Specific surface Area:6690cm^2/g
Expansive additive	EX	Ettringite-lime composite expansive additive, Density:3.05g/cm^3 Specific surface area:2840cm^2/g
Fine aggregate	S	Crushed sand, Density:2.61g/cm^3, Water absorption rate:1.07%
Water	W	Water supply
Water-reducing additive	SP	Superplasticizer, Polycarboxylate type

TABLE II
DETAILS OF MORTAR MIXTURES

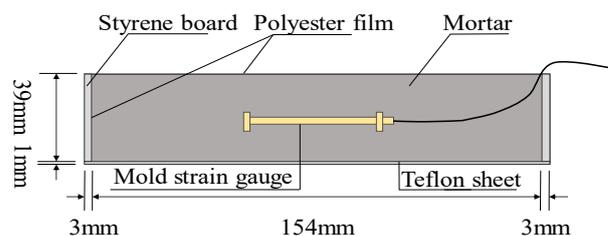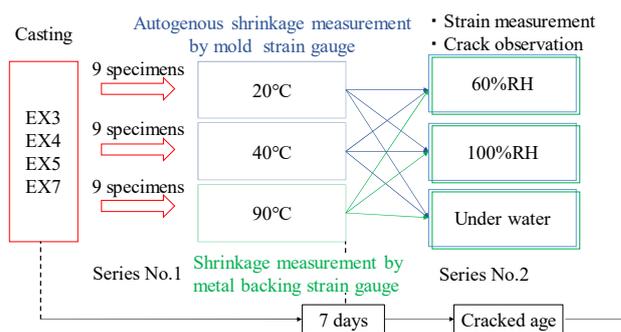| Symbol	W/B (%)	EX/B (%)	S (Vol%)	SP (%)
EX3		3		
EX4	16.5	4	30	3.1
EX5		5		
EX7		7		

Fig. 1 Diagram of specimen for shrinkage measurement

Fig. 2 Flow chart of experiment

B. Experiments on Autogenous Shrinkage (Series No.1)

In the first seven days of curing, a uniaxial length variation measurement of the mortar was carried out using mold strain gauges (Series PMF, applicable temperature range from -20 ℃ to 60 ℃), which were made from engineering plastic (Gauge length 50 mm). Styrene board of thickness 3 mm was placed at both ends of the 39 mm×40 mm×154 mm specimen. In order to eradicate the influence of friction forces with the formwork, a

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Teflon sheet (thickness 1 mm) was installed on the bottom of the specimen. In addition, to minimize the influence of evaporation and friction, the entire surface of the specimen was covered with polyester film. A diagram of the specimen is shown in Fig. 1.

After casting, the upper surface of the formwork was sealed with polyester film and aluminum tape. Nine specimens of each mixture were prepared, as shown in Fig. 1, and autogenous shrinkage was measured while curing at constant temperature circumstances of 20 °C and 40 °C. Respectively, specimens subjected to each curing temperature are marked with the suffix "-20" or "-40" in this paper.

Additionally, three specimens of each mixture were prepared without mold strain gauges installed and cured for seven days at a high temperature of 90 °C. These specimens are marked with the suffix "-90".

C. Experiments on Delayed Expansion (Series No.2)

On the 7th day, all specimens, cured under the conditions of 20 °C, 40 °C, and 90 °C, were removed from the mold after air-cooling in a thermostatic chamber at 20 °C. A metal base gauge (Series WFLM) was installed for the specimens cured at 90 °C. Afterwards, one specimen each of every mixture and curing temperature combination was placed in three different environments with relative humidity of 60% (60RH), 100% (100RH), and underwater (water), while their change in length over time was measured. Additionally, the appearance of the specimens was periodically observed to identify whether cracks occurred or not. The flow chart of the experiment is shown in Fig. 2.

D. Experiments on Hydration Analysis

Specimens sealed in aluminum bags were prepared and cured for seven days under constant temperature conditions of 20 °C and 40 °C for hydration analysis.

At the 7th day, the specimens in aluminum bags were broken into small pieces with a thickness of 5 mm or less, dipped in acetone for about 30 minutes, then separated from the acetone by suction filtration. Afterwards, the sample was again dipped in acetone for six hours and the same separation operation was carried out. Finally, the sample was dried for 24 hours in a desiccator before hydration analysis was performed. XRD/Rietveld analysis was performed using the D2 PHASER type XRD apparatus (Manufactured by BRUKER CO. LTD.), which obtained the components and contents of the hydrated product.

In addition, after long-term curing, one of the specimens for the length change test was crushed at a certain age and hydration analysis was conducted.

III. RESULTS AND DISCUSSION

A. Result of Autogenous Shrinkage Measurement

Figs. 3 and 4 show the autogenous shrinkage development within seven days at 20 °C and 40 °C, respectively.
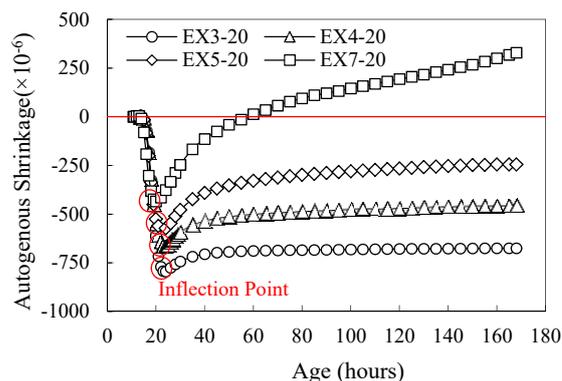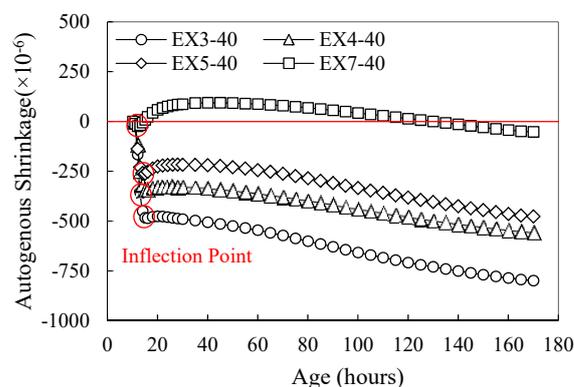

Fig. 3 Autogenous shrinkage variation with time (20 ℃)

Fig. 4 Autogenous shrinkage variation with time (40 ℃)

TABLE III
AUTOGENOUS SHRINKAGE OF INFLECTION POINT (× 10⁻⁶)

	EX3	EX4	EX5	EX7
20℃	-795	-641	-560	-435
40℃	-485	-315	-229	-23

The results of this experiment indicate that the rate of progress of autogenous shrinkage rapidly changes after one day, which was called "Inflection Point" (Marked with red circle in Figs. 3 and 4) by the authors. The autogenous shrinkage at the inflection point is shown in Table III. From Table III, it was found that the autogenous shrinkage at the inflection point of the specimens cured at 40℃ was smaller than that of the specimens cured at 20℃, for all proportions of expansion additive.

In Figs. 3 and 4, the expansion tendency in either temperature condition immediately after the inflection point is shown. However, shortly after 30 hours, specimens curing at 40 °C tend to start shrinking, whereas the specimens curing at 20 °C continued to expand regardless of the mixture proportion.

The autogenous shrinkage value observed at the age of seven days appears not to depend strongly on the temperature condition, but becomes larger as the proportion of expansive additive becomes larger. In particular, it was found that by adding 7% expansive additive by binding agent mass, and curing at 40°C, it is possible to almost entirely suppress the autogenous shrinkage before the seven-day mark. From the results, it was confirmed that autogenous shrinkage reduction in

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the early age can be improved by the addition of a large amount of expansive additive.

B. Production of Calcium Hydroxide at 7th Day

According to previous literature, it was confirmed that the autogenous shrinkage reduction effect of expansive additives is affected by the production of CH for high strength cement paste in the early age [2]. In this study, the amount of CH was obtained for specimens at the age of seven days by XRD/Rietveld analysis, the relationship between the production of CH and addition ratio of EX was summarized and shown in Fig. 5.
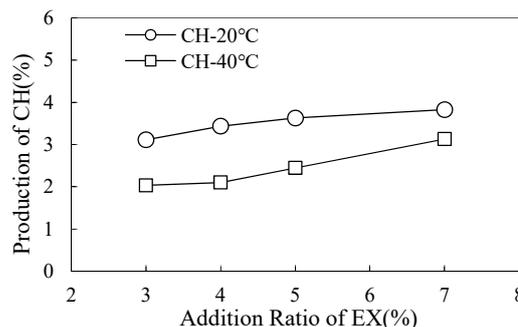
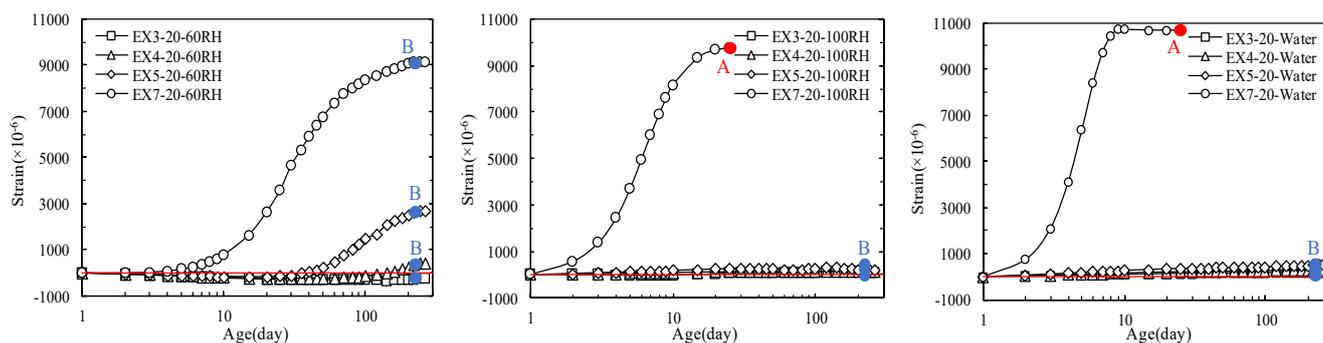Fig. 5 Production of CH variation with addition ratio of EX

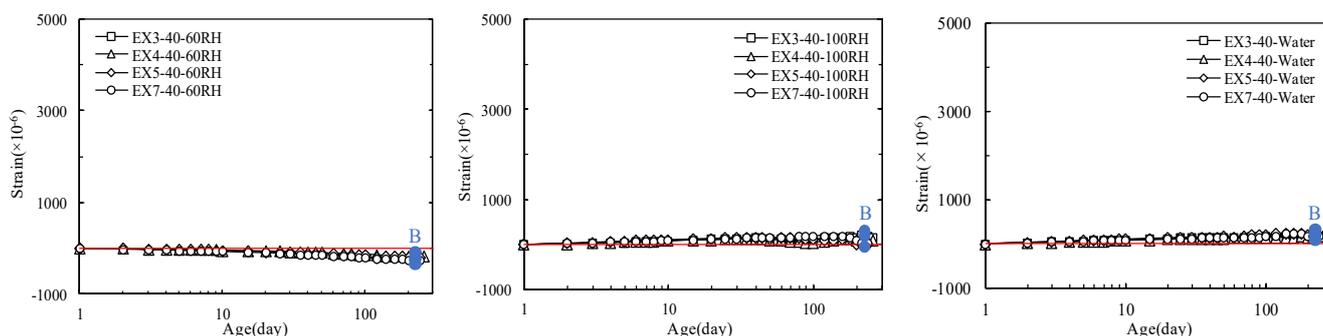Fig. 6 Strain variation with time (20 °C)

Fig. 7 Strain variation with time (40 °C)

As shown in Fig. 5, it was observed that regardless of the temperature condition, the production of CH at seven days increased as the ratio of EX increased. It was confirmed that increasing the proportion of EX will increase the production of CH, leading to autogenous shrinkage being effectively reduced in the early age.

Focusing on the temperature condition, it was found that the amount of CH at 7 days was higher when curing at 20°C than that of when curing at 40°C, even when the same amount of EX was added. It is well known that, for the silica fume premixed cement, the pozzolanic reaction of silica fume consumes CH, and according to the study of Kamikoshi, the pozzolanic reaction was found to be activated by an increased curing temperature [12], additionally the reaction accelerates as the curing temperature is further increased [13]. Consequently, more CH was consumed by the pozzolanic reaction when curing at the temperature of 40 °C.

C. Strain after 7 Days

After curing at 20 °C, 40 °C, and 90 °C for seven days, strain measurements of all the mortar specimens were taken while curing in an environment of 20 °C and relative humidity of 60%, 100%, or underwater, for a long time. The strain variation with time after seven days is shown in Figs. 6 and 7 with the origin representing the condition at seven days.

In Fig. 6, after initial curing at 20 °C, EX7-20-60RH at 13 days, as well as EX7-20-100RH and EX7-20-Water at eight days, show significant expansion strain. According to the previous research, when using a large amount of expansive additive, the occurrence of harmful delayed expansion was reproduced regardless of the curing condition. In addition, for the same curing condition, the delayed expansion tended to occur faster as the proportion of expansive additive increased.

Specimens where large expansion was confirmed by the strain measurement, were removed from the curing tank on the 36th day and the appearance was observed. All three specimens

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showed expansion cracks. The appearance of the specimens is shown in Fig. 8, where shell-like swelling cracks occurring in the entire specimen, causing typical expansion degradation. However, the specimens of EX7-20-60RH, which have a strain value of 9000 ($\times10^{-6}$) at the age of 260 days, were not found to have cracks. Similar expansion tendencies are shown in EX5-20-60RH and EX4-20-60RH, in which the proportion of the expansive additive is smaller than EX7. The occurrence of cracks before this time was not observed, but it is possible that this could lead to a large expansion strain as the age of the specimen increases.

Considering the initial curing temperature, for 20°C, there was a certain degree of expansion tendency under any humidity circumstance when 7% of expansive additive was added, whereas for 40 °C, there is almost no expansion tendency until the age of 250 days, no matter what the humidity condition was. From these results, it was inferred that most of the expansive additive was reacted when curing took place in an initial high-temperature of 40 °C, whereas some expansive additive remained after curing at 20 °C, after the first week, the remaining expansive additive gently reacts and causes expansion during long-term curing.

D. Content of Hydrates after 7 Days

In order to ascertain the cause of delayed expansion due to expansive additives, the content of hydrates of a certain age was also investigated.

For specimens of EX7-20-Water and EX7-20-100RH in which remarkable cracking was observed, all three specimens were crushed on the day (Marked 'A' in Fig. 6) when occurrence of cracking was confirmed, hydration was stopped, and the content of expandable hydrates CH and AFt were obtained by XRD/Rietveld analysis. Additionally, one of the three specimens that cured in 60% RH was crushed after 220 days of curing (Marked 'B' in Fig. 6), when large expansion strain was confirmed, and the content of CH and AFt were obtained using the same method. The result for specimens of EX7-20 is shown in Fig. 9, and the production at seven days is indicated by a dotted line.

Fig. 9 shows that for EX7-20-Water, both the content of CH and AFt has a large value that is significantly bigger than the seven day value. For EX7-20-100RH, both CH and AFt show almost the same content value as they did at seven days. On the other hand, the result of EX7-20-60RH shows a relatively small content value of CH but nearly the same value of AFt when compared with the seven day values. The decrease in CH is attributed to the pozzolanic reaction of silica fume, which consumes more CH than is produced by the hydration of EX in long-term curing. The results lead us to believe that the expansion destruction of EX7-20-Water and EX7-20-100RH is due to the excessive formation of CH and AFt at around 36 days of curing.

For comparison, the same hydration analysis was performed on a specimen of each compound that cured under each circumstance for around 220 days (Marked 'B' in Figs. 6 and 7). The results are shown in Figs. 10 and 11. The content of hydrate obtained at the age of seven days is again indicated by a
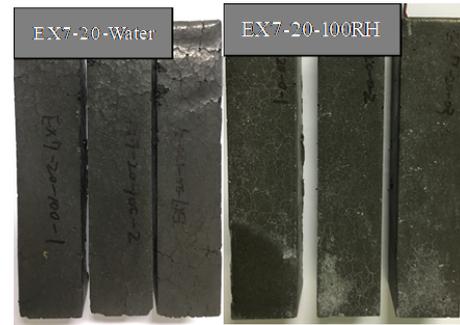
dotted line.

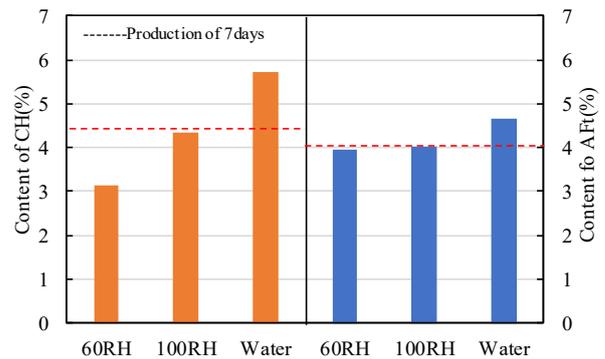Fig. 8 Appearance of delayed expansion cracks

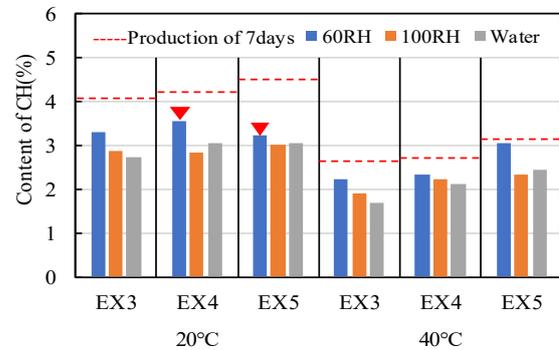Fig. 9 Content of expandable hydrates for EX7-20 after long-term curing

Fig. 10 Content of calcium hydroxide for specimens curing under each circumstance in 220 days
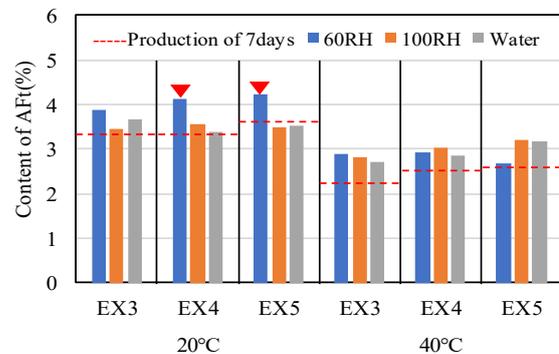

Fig. 11 Content of ettringite for specimens curing under each circumstance in 220 days

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As shown in Fig. 10, regardless of the initial temperature condition or the curing humidity, the content of CH in all specimens of each compound was decreased, when compared with the production at the 7th day. This is a result of the pozzolanic reaction, as the speed of consumption for CH is faster than the production when relatively less EX (EX3, EX4, EX5) is added. At 220 days, the specimens of EX4-20-60RH or EX5-20-60RH (marked with red triangle in Fig. 10) did not have a significant larger value of CH content than others, yet did show expansion tendency, as seen in Fig. 6.

From Fig. 11, it was confirmed that all the specimens show a lager content of AFt than seven-day values, and especially for the specimens with the compound of EX4-60RH and EX5-60RH (marked with red triangle in Fig. 11), which show a significantly larger content of AFt than others when curing at 20 °C in the early age. This corresponds to the results in Fig. 6 (upper left) that show that large expansion strain appears in the specimens curing under the circumstance of 60%RH for a long time.

The considerations above lead us to believe that for the specimens of EX7-20-100RH and EX-20-Water in the relatively early age, expansion deterioration occurred due to the excessive content of both CH and AFt, whereas the large expansion occurred in EX7-20-60RH only due to a large amount of AFt. There is a possibility that the hydration product C-S-H was changed by drying and the movement of sulfate ions was promoted, causing this difference, but the mechanism of which should be investigated in the future.

Furthermore, the crystallization stress theory, which is summarized as "the ettringite will form under the supersaturation and result in the crystallization pressure", can be used to explain the expansion due to the formation of ettringite [14]. From previous studies [15]-[17], it is necessary to consider the conditions of crystal volume, saturation degree, and pore solution when using the crystallization stress theory to explain the expansion pressure. There is a lack of data in this paper to provide further discussion on this subject but it should be addressed in future studies.

IV. CONCLUSION

In this study, the effect of expansive additives in ultra-high strength mortar on autogenous shrinkage and delayed expansion was investigated. The ratio of expansive additive was varied in the mortar samples, strain variation with time of the mortar specimens under different curing conditions was measured, and hydration analysis was conducted, from which the following conclusions was obtained.

(1) In the early age, autogenous shrinkage of ultra-high strength mortar can be effectively reduced by adding more expansive additive in a certain ratio. It is possible to obtain a non-shrinking state by adding 7% expansive additive by binding agent mass, and curing at a constant temperature of 40°C for 7 days.

(2) It was confirmed that the more expansive additive that is added, the more calcium hydroxide is produced causing the autogenous shrinkage reduction effect to improve for the first seven days.

(3) The mortar specimens which contain 7% of expansive additive by binding agent mass, and are cured at an initial constant temperature of 20 °C, experience large expansion strain after seven days of curing regardless of curing humidity. For the specimens cured underwater and in moist air, the occurrence of delayed expansion cracks was confirmed.

(4) It was inferred that, in ultra-high strength mortar the formation of a large amount of calcium hydroxide and ettringite at an early age due to the addition of expansive additive contributes to the occurrence of delayed expansion, whereas the occurrence of large expansion strain is only due to the formation of ettringite as the curing age increases.

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