STUDY ON THE COMBINED ALKALI SILICA REACTION AND DELAYED ETTRINGITE FORMATION

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ABSTRACT: While expansive reaction due to the Alkali-Silica Reaction (ASR) and Delayed Ettringite Formation (DEF) have been found to be correlated and the appearances of their cracking patterns are similar, the dominance of DEF or ASR on the expansion is still unknown. However, it is generally believed that ASR and DEF cause crack formation leading to a deleterious effect on concrete. This study investigated the expansion due to ASR, DEF and their combination coupled with the dynamic Young's modulus (E_D) and water absorption in concrete test specimens, some of which contained reactive aggregates through a replacement of 30 wt% of the coarse aggregate. K_2SO_4 and $CaSO_4$ were further added (2.5 wt% SO_3 addition) to study the effect of reactive metal ions with sulphate ion on ASR and DEF. Some specimens were cured at an elevated temperature of 90 °C at the early age to accelerate the DEF expansion. The specimens with the addition of SO_3 showed higher expansion and lower E_D as compared to those without SO_3, regardless of the curing temperature. The expansion was significant and rapid with the reduction of E_D when reactive aggregate and K_2SO_4 were used. According to the expansion with E_D and moisture absorption, it is indicated that ASR may promote DEF to cause the expansion and subsequent damage after hardening when adequate sulphate and a high temperature are concurrently present.

Keywords: Expansion, Alkali-Silica Reaction, Delayed Ettringite Formation, Dynamic Young's Modulus

1. INTRODUCTION

Deterioration in mass of concrete structures is typically ascribed to thermal deformation or shrinkage. However, from onsite inspections, severe map cracking, which appears to be different from typical cracking, has been found on the surfaces of concrete structures, such as the bridge foundation in Thailand shown in Fig. 1. Such crack patterns can generally be considered to result from the Alkali-Silica Reaction (ASR). Furthermore, some authors [1,2] suggest that Delayed Ettringite Formation (DEF) occurring in heat-steam cured concrete and massive concrete structures where the internal temperature exceeds 70 °C may additionally play a role.

Although there have been many studies on the expansion associated with ASR and DEF, the primary cause of damage is still controversial. While the mechanisms behind ASR and DEF differ, the phenomena are correlated and the appearance of their cracking patterns are similar. Biangam [3] examined the core concrete extracted from existing concrete structures that were constructed over twenty years ago in Thailand. It was suggested that ASR gel and ettringite were observed in many locations such as at the rim around aggregates, in voids and in cracks of paste and aggregates while DEF symptoms were more visible at a 100 cm depth in the concrete. A similar investigation was made

Fig.1 Cracking of a concrete foundation in Thailand

by Jensen [4] to suggest possible DEF and the occurrence of ASR in a concrete foundation in Thailand. They suggested that DEF occurs first and followed by ASR. This might be because the aggregates have been activated to be alkali reactive by high temperature, even though Thai cements generally have low alkali content (Na$_2$O eq. varying from 0.2% to 0.48%).

Thomas [5] investigated damaged concrete columns of an expressway in North America, and implicated that both ASR and DEF are the possible causes of concrete deterioration. In one column, DEF was the sole contributor to deterioration with no evidence of any contribution from ASR. In contrast, according to Diamond [6], the association of DEF with prior cracking induced by ASR is not accidental, and instead may be fundamental to the

DEF process. Ettringite formation is retarded in pore solutions that have a high concentration of alkali hydroxide, such as for high alkali cement. Since ASR progressively reduces the alkali hydroxide concentration, this investigator postulated that such a reduction might trigger ettringite precipitation. However, the primary cause of damage, whether DEF or ASR, is still uncertain. This becomes more ambiguous in actual structures, as at the time of investigation of the concrete sample [3-5], which has been in service for over ten years, both ASR and DEF may have already occurred

For laboratory tests with the occurrence of ASR or DEF or both, in which the effect of the cement composition, the temperature of curing, and the addition of SO_3 or submerged conditions were studied, there are some contradictory results on the rate of expansion and final expansion. Shayan [7] concluded that ASR is the primary cause of expansion, as a steam-cured specimen without reactive aggregate did not show any sign of DEF even in the presence of excess $CaSO_4$ while DEF expansion appeared when reactive aggregate was used. This conclusion is contradictory to that of Kelham [8]. Kelham suggested that mortars cured at 90 °C with raised $CaSO_4$ content caused a mild expansion even without reactive aggregates, while K_2SO_4 caused a significant expansion when used to obtain an equal sulphate content. Later, Shayan [9] also investigated the effect of many parameters such as sulphate, alkali content in various cements and curing temperature on the expansion of mortar and concrete. It was concluded that the most deleterious expansion is associated with ASR arising from high alkali content and reactive aggregates while DEF may occur only when the relevant factors including alkali, sulphate, aluminate, and high temperature (around 85 °C) are concurrently present, regardless of the reactive aggregates. Similar discrepancies were also investigated by Hanehara [10] who studied the relationship between DEF and ASR in mortar bars with two different sulphate salts: K_2SO_4 and $CaSO_4$. Although further research was recommended to verify their conclusion, they concluded that DEF could be caused by the addition of $CaSO_4$ when the concrete is subjected to high-temperature steam. While $CaSO_4$ does not provide alkalis for ASR in contrast to K_2SO_4, it can still supply sulphate, which might contribute to the DEF expansion.

The objective of this study is to investigate and confirm the effect of ASR and DEF on expansion and attendant damage in concrete focusing on the influence of SO_3 with varying chemicals to add sulphate ions to the pore solution. Coarse reactive aggregates were used to initiate ASR. The internal damage due to the expansion was characterized through correlation with the dynamic Young's modulus. The mass gain with expansion was investigated focusing on the water absorption by the ASR gel, the formation of ettringite, and water penetration into the internal pores.

2. EXPERIMENTAL PROGRAM

2.1 Materials

Ordinary Portland Cement (OPC) was used to cast the specimen as it is widely used for precast concrete and massive concrete with a high heat of hydration. The chemical composition of the OPC is given in Table 1. The fine aggregate was river sand with a density of 2.62 g/cm^3 in saturated surface-dry conditions. Sandstone with a density of 2.66 g/cm^3 and a maximum size of 20 mm was used as a non-reactive coarse aggregate to satisfy Japanese Industrial Standards (JIS). The reactive aggregate used in this specimen is highly reactive andesite containing opal and cristobalite volcanic ash with a density of 2.52 g/cm^3 [11].

To accelerate the DEF, SO_3 (2.5 wt% of cement) was added to increase sulphate content in the mix proportion using K_2SO_4 and $CaSO_4 \cdot 2H_2O$ (hereafter, $CaSO_4$). Two types of sulphate salts were used to examine the influence of different reactive metal ions on ASR and DEF.

Table 1 The chemical composition of the OPC used in this study.

Chemical composition	% by weight
Loss on Ignition (ig. loss)	2.18
Silicon Dioxide (SiO_2)	20.45
Aluminum Oxide (Al_2O_3)	5.15
Iron Oxide (Fe_2O_3)	3.02
Calcium Oxide (CaO)	63.50
Magnesium Oxide (MgO)	2.34
Sulphur Trioxide (SO_3)	1.95
Sodium Oxide (Na_2O)	0.18
Potassium Oxide (K_2O)	0.35
Titanium Dioxide (TiO_2)	0.28

2.2 Specimens and Proportion

Concrete specimens were prepared with the mixture proportions as shown in Table 2. Each mixture had a water-cement ratio of 0.5 and a ratio of fine aggregate to the total aggregate of 47.24% by weight. Concrete was cast in a 100 x 100 x 400 mm prism using a specially designed steel mould with a system to insert steel studs at both ends of the specimen for measurement of the post-hardening length change.

To ensure the occurrence of ASR, a set of specimens were cast by replacing 30 wt% of coarse aggregate with the reactive aggregate.

To accelerate the DEF effectively, the temperature history was adapted based on the work

Table 2 Mixture proportions of the test specimens.

Mix proportion	Type of sulphate addition	C (kg)	W (kg)	Sand (kg)	NR (kg)	R (kg)	K_2SO_4 (kg)	$CaSO_4$ (kg)
NSR	-	348	174	770	602	258	-	-
SNR	K_2SO_4	348	174	770	860	-	18.9	-
SNR	$CaSO_4$	348	174	770	860	-	-	18.7
SR	K_2SO_4	348	174	770	602	258	18.9	-
SR	$CaSO_4$	348	174	770	602	258	-	18.7

Note: S = Addition of Sulphate, NS = Non-Addition of Sulphate
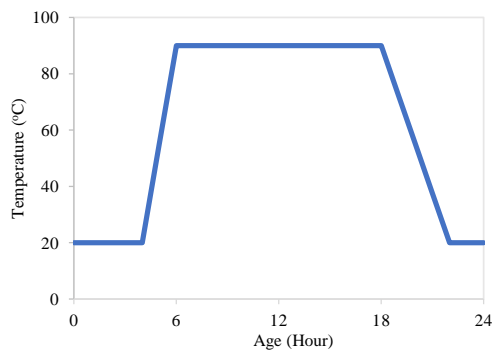R = Reactive aggregate, NR = Non-reactive aggregate

Fig.2 Early age temperature history to accelerate DEF

of Famy [12,13] as shown in Fig. 2. A set of specimens (suffixed by _90) were mould cured with sealing on the top surface for four hours under a sealed condition at 20 °C soon after casting, and then exposed to 90 °C for twelve hours with a temperature gradient of 35 °C/hr. Subsequently, the temperature was gradually reduced from 90 °C to 20 °C over four hours, the specimens were kept at 20 °C for another two hours and then finally the seal and mould of the specimens were removed. Another set of specimens, suffixed by _20, were cured at 20 °C for twenty-four hours under the sealed condition. After mould removal, all specimens were submerged in tap water and stored in a climate-controlled room at 20 °C and 60% relative humidity for twenty-four hours. The specimens were then removed from the water and air-dried about two hours before measure the initial length of the specimen. As the DEF expansion rate may be retarded when the pH in the pore solution rises due to leaching [13], the water used to submerge the specimens was replaced every month.

2.3 Expansion, Weight, and Dynamic Young's Modulus Measurements

The length change was measured by a dial gauge with a precision of 0.001 mm and a base length of 420 mm. The weight was measured with a precision

of 1 g. The measured values of three specimens for each type of mix proportion and curing condition were then averaged.

The dynamic Young's modulus was measured using a non-destructive method and the primary resonance frequency as measured using the frequency analyser (model number MIN-011-0-10, serial no. 09126). The dynamic Young's Modulus is then calculated by Eq. (1). The dynamic Young's modulus measurement complied with ASTM E1876-01.

$$E_D = 9.47 x 10^{-4} \frac{L^3 T}{bt^3} mf_1^2 \tag{1}$$

where E_D is the dynamic Young's modulus (N/mm²), L is the length (mm), T is the coefficient determined by K and L, b is the section length (mm), t is the vibration direction length (mm), m is the mass (kg) and f is the primary resonance frequency (Hz).

The mass gain was calculated as a percentage by using the relation

$$\% \text{ Massgain} = \left(\frac{W_D - W_0}{W_0} \right) x 100 \tag{2}$$

where W_D is the measured mass at time of measurement (g) and W_0 is the initial mass measured before submerging the specimens in tap water (g).

3. RESULTS AND DISCUSSION ON EXPANSION

Fig. 3 shows the evolution of the expansion of various specimens with elapsed time after submerging the specimens in tap water. The specimens are denoted by the type of sulphate addition (K for the addition of K_2SO_4 and Ca for the addition of $CaSO_4$) followed by the addition of sulphate, the use of reactive aggregate, and curing temperature. For example, the specimen with a reactive aggregate containing SO_3 from K_2SO_4 and cured at 90 °C is denoted as "K_SR_90." The specimens with K_2SO_4 and $CaSO_4$ were cast on
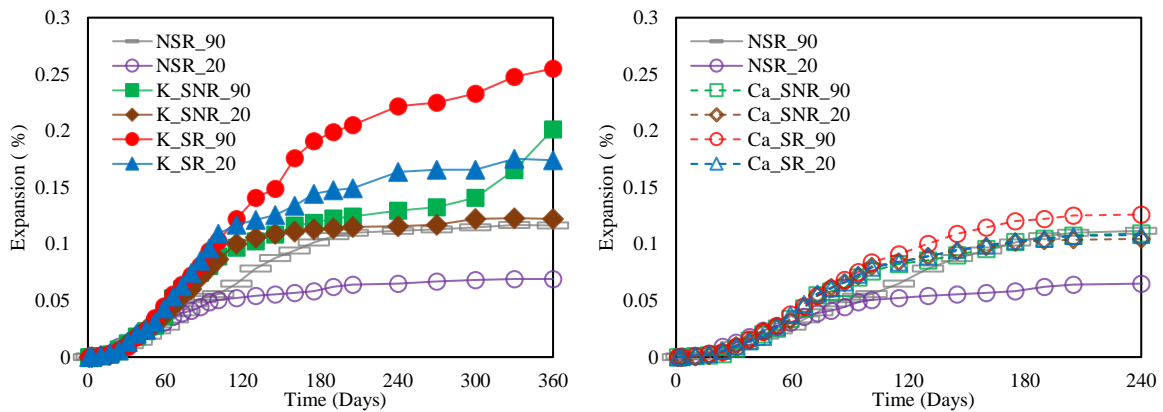
Fig.3 Expansion variation with time of the concrete specimens

different dates (the Ca series was started later); the most recent data is reported here.

Overall, the concrete specimens with the addition of SO_3 for both K_2SO_4 and $CaSO_4$ demonstrated more expansion than those without the added sulphate. The expansion is more significant for specimens cured at elevated temperatures rather than those at 20 °C with the same mix containing the addition of SO_3. For the same mix proportions, similar expansion trends are observed whether K_2SO_4 or $CaSO_4$ is added, while the expansion of the specimens with the added K_2SO_4 had larger and more rapid expansion. The specimen Ca_SR_90 showed more expansion than Ca_SNR_90, indicating that the expansion of the specimens cured at elevated temperatures is significantly enhanced in the presence of a reactive aggregate. Moreover, K_SNR_90 showed more expansion than K_SR_20, suggesting that DEF causes more expansion at later stage under certain conditions than ASR. For the highest expansion in the specimens coupled with ASR and DEF (K_SR_90), there is a larger overall expansion value than either individual effect. In the case of added $CaSO_4$, the start of Ca_SR_90 and Ca_SR_20 expansion is delayed by approximately 240 days as compared to the addition of K_2SO_4.

3.1 The Effect of Added Metal Sulphate Type on Expansion

It was found that the specimens with K_2SO_4 with or without reactive aggregate, (K_SR, K_SNR), showed a considerably larger expansion over specimens without the added K_2SO_4 (NSR). The addition of K_2SO_4 causes much higher expansion than the addition of $CaSO_4$ (approximately 1.9-2.2 times at 240 days). Although there is a difference in the magnitude of expansion, the expansion trend of specimens with a high temperature history and containing reactive aggregate is similar. In the case of added K_2SO_4, the dissolved sulphate ions are partially consumed to form ettringite, allowing the

potassium ions to act as catalyser to break the silica bonds of reactive aggregates [14], thus resulting in ASR expansion. Alternatively, $CaSO_4$ is an alkaline-earth sulphate that would not provide alkali ions for ASR as the alkaline-metal sulphate (K_2SO_4) does, while it instead may contribute to the DEF expansion since it consists of sulphate ions. The addition of $CaSO_4$ to a concrete subjected to steam curing at high temperatures should in principal increase the risk of occurrence of a large expansion due to DEF according to previous studies [10]; The effects of $CaSO_4$ on the expansion was not observed in our result even though it was exposed to high temperature which is contradictory to Hanehara and Oyamada [10] who suggested that DEF could be caused by the addition of $CaSO_4$. A reason to support this difference result might be due to the type of cement. High Performance Cement (HPC) was used in Hanehara and Oyamada's work [10], while Ordinary Portland Cement (OPC) was used in our study. From this perspective, it can be suggested that higher SO_3 in HPC can be considered as the important factor that might lead to more considerable expansion than the lower SO_3 in OPC. Zhang [15] reported that significant expansion was observed for mortars made from cement with SO_3/Al_2O_3 molar ratios between approximately 0.85 and 1.4 and with high temperatures at early ages. In the present study, the total SO_3 in the cement including external additions was approximately 4.5 wt% giving a molar ratio of SO_3/Al_2O_3 of 1.10, which is above the range given by Zhang et al. [15]. A significant expansion was observed in the specimens K_SR_90 and Ca_SR_90, where the expansion was approximately 0.25% and 0.13% at 360 and 240 days, respectively. The expansion of K_SNR_90 rises suddenly at approximately 330 days probably due to the occurrence of DEF. Alternatively, Ca_SNR_90 did not yet exhibit a large expansion, which suggests that DEF may not significantly
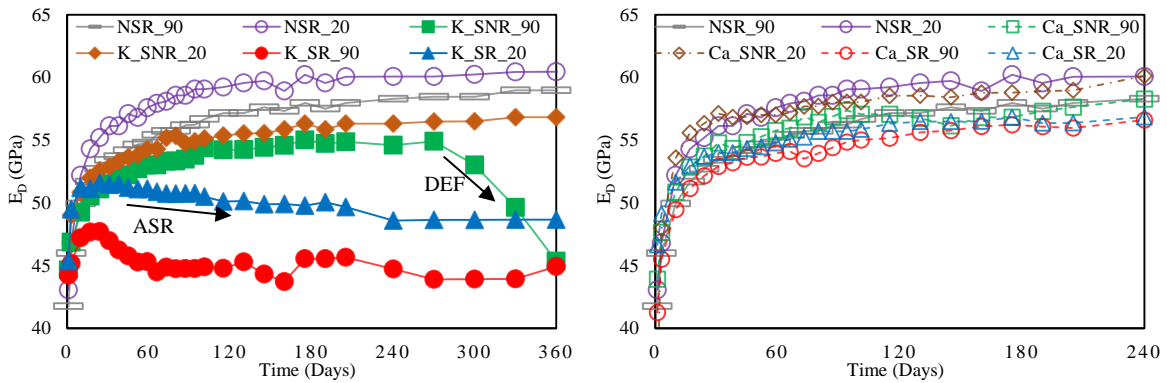
Fig. 4 Variation in the dynamic Young's modulus

occur even with available sulphate ions.

It was observed that even when the concrete was not subjected to a high temperature, a gradual expansion occurred. K_SR_20 showed more expansion than Ca_SR_20, which could be attributed to ASR owing to an increased potassium concentration from the added K_2SO_4, as calcium does not provide alkalis for ASR unlike potassium.

Ca_SR_20, Ca_SNR_20 and K_SNR_20 had similar expansions. It can be suggested that small amounts of ettringite might form even at lower temperatures when sufficient sulphate ions are available.

3.2 The Effect of Reactive Aggregate on Expansion

In the absence of reactive aggregates and excess SO_3 (specimens K_SNR_90 and K_SNR_20), the expansion was lower as compared to specimens with reactive aggregates (K_SR_90 and K_SR_20) at the same temperature exposure. At initial stage, the expansion of K_SR_20 is higher than K_SNR_90, indicating that ASR may occur with reactive aggregate and K_2SO_4 addition regardless of temperature history and causes a more rapid expansion of the concrete than DEF. According to Diamond [16], the addition of alkali dissolved in the mixing solution can increase the sulphate concentration. Thus, the specimen K_SR_20 reveals the essential factors that are necessary for the expansion process in the absence of high temperatures: reactive aggregate and a considerable concentration of alkali from potassium and sulphate in the concrete pore solution. When ASR somehow experimentally occurs even over short time periods, the pH in the pore solution would be reduced to promote the expansive ettringite formation in the hardened cement paste even though a high temperature is not supplied at an early age.

In the case of added $CaSO_4$, the expansion of Ca_SR_20 and Ca_SNR_90 was approximately equal to 0.11% at 240 days. This might be because calcium does not cause ASR in Ca_SR_20 while

DEF has not yet occurred in Ca_SNR_90.

3.3 The Effect of Curing Temperature on Expansion

The expansion was higher in the presence of a reactive aggregate and added sulphate (K_SR_90 and Ca_SR_90), which might be due to the high curing temperature initiating ASR, while the subsequent reaction takes place later in moist curing conditions [17]. In the absence of a reactive aggregate, the larger expansion of K_SNR_90 than K_SNR_20 at 360 days was likely due to ettringite formation at a later age. Normally, when concrete has added sulphate, alkali content, and is exposed to a high temperature (over 70 °C) at an early stage, DEF is more likely to occur [18]. A continued expansion even at 360 days is evident from the DEF requiring more time to complete the reaction [9]. In the case of added $CaSO_4$, the expansions of Ca_SNR_90 and Ca_SNR_20 were similar. This might be due to DEF in Ca_SNR_90 not yet occurring at 240 days. The expansion in the case of added $CaSO_4$ requires more time before observation.

4. EVOLUTION OF THE DYNAMIC YOUNG'S MODULUS WITH TIME

Fig. 4 shows the overall dynamic Young's modulus (E_D) for the concrete prisms at various elapsed times after submerging the specimens into tap water. The initial E_D varies from 40-45 GPa based on the type of SO_3 addition and the curing condition. The specimens cured at the elevated temperature show a higher E_D for the first seven days. This is due to the accelerated hydration reactions from the high curing temperature that can cause the concrete to have early strength development yet consequently gain less strength development at later stages [19].

Considering the type of SO_3 addition, Ca_SR_90 has a higher gain in E_D (without a gradual decrease of E_D) than K_SR_90 at 100 days.

Table 3 Observation time of visible cracking.

Proportion	Days
K_SR_90	210
K_SR_20	270
K_SNR_90	330
Others	Not observed

This is because while calcium sulphate does not provide alkalis unlike potassium sulphate, the DEF requires more reaction time for Ca_SR_90.

There is a possibility that the E_D in the specimen cured at 20 °C with added K_2SO_4 (K_SR_20) can be gradually reduced due to the internal damage by ASR. The ASR gel degrades the modulus of the concrete long before creating visible cracking on the surface, with few surface cracks visible at 360 days. The time at which visible cracking was observed is given in Table 3.
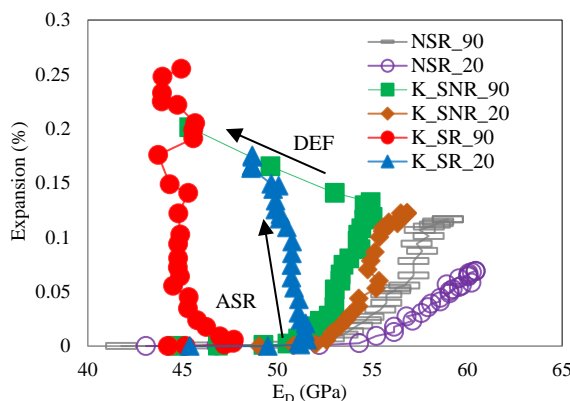
In the specimens with only the acceleration of DEF (K_SNR_90), the E_D gradually increases yet falls sharply at the age of 270 days, which might be contributable to DEF at the later stage. The DEF expansion might cause sudden damage, resulting in a sharp reduction of E_D.

In the specimens coupled with ASR and DEF (K_SR_90), the E_D increases at first and then gradually decreases after reaching a peak at approximately 20 days. Subsequently, the E_D falls sharply (at approximately 100 days), though not as rapidly as for K_SNR_90. Internal cracking likely occurred due to ASR gel formation (at approximately 30-100 days), and later ettringite formation may form preferentially in crack zones [20] undergoing ASR is not a reason to cause the expansion with large reduction of E_D.

The relationship between the modulus and the expansion is discussed in the next section.

5. RELATIONSHIP BETWEEN EXPANSION AND THE DYNAMIC YOUNG'S MODULUS

Fig. 5 shows the relationship between expansion and the dynamic Young's modulus for various specimens. Initially, E_D increases rapidly by approximately 4-5 GPa without noticeable expansion, which might be due to a hydration reaction in the early stage improving strength development; however, at the later stage when the expansion becomes larger, the dynamic Young's modulus was retarded and reduced. This is in agreement with Zhang [15] that the dynamic Young's modulus degrades when the expansion exceeds 0.15%. A reduction began even for small expansions as shown by the K_SR_90, K_SR_20, and K_SNR_90 specimens.

Variations were observed between specimens with added K_2SO_4 and $CaSO_4$. The expansion and the modulus show concurrent development when $CaSO_4$ was added, while the modulus of the specimen with added K_2SO_4 was retarded and reduced when the expansion became larger. The reason for these different tendencies is probably due to the potential combination of reactive silica and alkali content from K_2SO_4 that may produce a deterioration as seen in K_SR_90 and K_SR_20.

In the specimens that exhibited a large expansion, there was a steeper slope from a gradual degradation of E_D in K_SR_20 and K_SR_90 compared to the slope of K_SNR_90 that increased to the right hand side at the beginning and rapidly reduced to the left hand side at a later age. The gradual degradation in K_SR_20 indicates that K_2SO_4 and reactive aggregate causes ASR expansion and subsequent damage at the initial stage due to ASR gel that produce an internal tensile strength and easily affect to ASR as a reduction of the modulus [21]. In the specimens coupled with ASR and DEF (K_SR_90), the ASR acceleration at the initial stage from the elevated temperature can lead to an immediately degradation of dynamic Young's modulus and early expansion. The subsequent damage and rapid expansion at later stages might be caused by DEF, which likely results from a lowered pH by ASR occurrence.

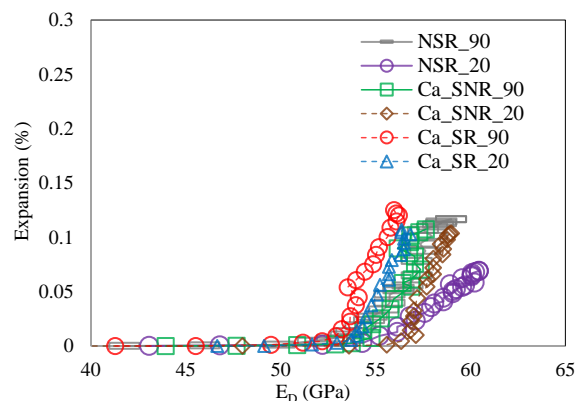Alternatively, the increased slope for

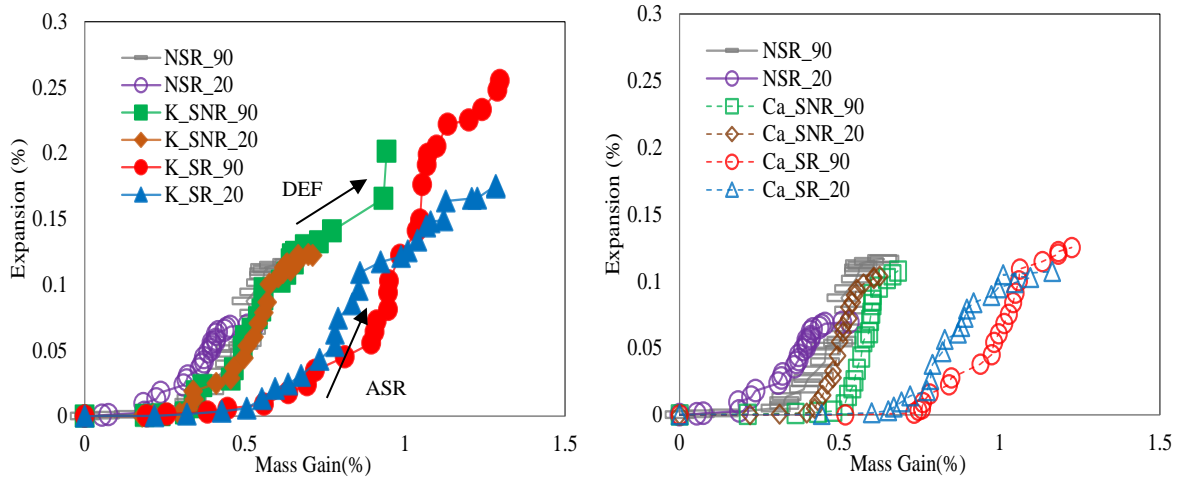Fig.5 Relationship between expansion and the dynamic Young's modulus

Fig.6 Relationship between expansion and mass gain

K_SNR_90 might be due to the accelerated hydration from an increased curing temperature as aforementioned [19]. At later stages, the slope of K_SNR_90 starts to fall sharply to the left hand side when the expansion reaches approximately 0.15%, which might be due to the occurrence of DEF leading to a rapid reduction of the modulus.

6. RELATIONSHIP BETWEEN EXPANSION AND MASS GAIN

Fig. 6 shows the relationship between the expansion and the mass gain for each specimen. Similar tendencies are observed for the K_2SO_4 and $CaSO_4$ specimens, even though the specimens with added K_2SO_4 showed a larger expansion. However, before the expansion becomes larger, the mass gain of specimens with added $CaSO_4$ was higher than for added K_2SO_4, which might be related to ettringite formation in the early hydration stage from gypsum reaction with C_3A preventing expansion.

In the absence of reactive aggregate with added SO_3, the specimens cured at an elevated temperature (K_SNR_90, Ca_SNR_90) had an accelerated mass gain, especially after the onset of expansion. This might be due to the combined effect of ettringite formation and moisture absorption during the expansion process. Moreover, the steeper slope of the mass gain of specimen K_SR_20 as compared to K_SNR_90 indicate that water absorption by the ASR gel might be the primary cause of expansion and mass gain.

In the specimens coupled with ASR and DEF (K_SR_90) the mass increases gradually at first and then rapidly when the expansion became larger. This indicates that the ASR gel absorbs moisture at the initial stage, and at later stages, the occurrence of DEF with the crystalline water in ettringite leads to further moisture absorption and expansion.

7. CONCLUSION

In the present study, the expansion due to the effect of sulphate, reactive aggregate, and curing temperature was investigated. The dominant factor in determining expansion and the dynamic Young's modulus is the addition of SO_3, with the effect becoming more prominent in the presence of a reactive aggregate (30wt% of coarse aggregate). Test specimens with added sulphate showed higher expansion than specimens without added sulphate (approximately 1.4-1.7 times). This experiment can be summarized as follows.

(1) The addition of 2.5 wt% SO_3 is a significant factor for increased expansion regardless of the initial temperature.
(2) The test specimens with the reactive aggregate showed higher expansion and a lower dynamic Young's modulus, and this effect was significantly enhanced when SO_3 was added.
(3) The mass of specimens increased with an increase in expansion. This effect was greater for specimens cured at an elevated temperature. Furthermore, the effect was enhanced in the specimens with added SO_3.
(4) The addition of K_2SO_4 gave a large, rapid expansion and a lower dynamic Young's modulus compared to the addition of $CaSO_4$.
(5) At initial stage, the expansion related to ASR causes more damage than the expansion due to the ettringite formation when acting independently. However, the expansion due to a combination of ASR and DEF causes much higher damage. ASR may occur first and later promote ettringite formation to cause the expansion and subsequent damage after hardening when adequate sulphate and a high temperature are concurrently present.

8. ACKNOWLEDGMENTS

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