Influence of maximum temperature at early age of concrete and R_2O and/or SO_3 content on DEF expansion of concrete

Shunsuke Hanehara [(1)], Tetsuya Oyamada [(2)]
(1) Faculty of Science and Engineering, Iwate University, Morioka, Japan, hanehara@iwate-u.ac.jp
(2) Faculty of Science and Engineering, Iwate University, Morioka, Japan, oyamada@iwate-u.ac.jp

Abstract

The occurrence of DEF is strongly affected by the maximum temperature of concrete at early age. The alkali content (R_2O: Na_2O equivalent), SO_3 content, and C_3A content also affect the occurrence of DEF depending on the conditions. In this study, the influence of independently added R_2O content and SO_3 content on DEF expansion was investigated assuming the mortar portion of concrete with unit cement content of 300 kg/m^3 (R_2O: 1.7-11.2 kg/m^3, SO_3: 9.6-20.6 kg/m^3). Two types of cement (High early strength Portland Cement: HPC and Ordinary Portland Cement: OPC) were used and the mortars were affected by high temperature at 70°C, 80°C, and 90°C.

The results obtained in this study closely agree with the content of the JCI Guidelines 2016 [3], thus supporting the proposal in these Guidelines of threshold values for the maximum temperature of concrete to prevent DEF cracking indicated. Regarding the range showing expansion at the maximum temperature of 70°C, when R_2O content is large, it is necessary to lower the threshold values of SO_3 content from about 17 kg/m^3 to about 15 kg/m^3.

When only HPC is used and the maximum temperature is 70-90°C, the expansion increases as the maximum temperature increases, compared with equal R_2O content and SO_3 content. If the R_2O content and SO_3 content are small, no expansion occurs.

When only HPC is used and the maximum temperature is 90°C, the maximum value of expansion tends to increase as R_2O content and SO_3 content increases, as in previous studies.

Comparing the two types of cement, HPC and OPC, HPC starts expanding with less R_2O content and SO_3 content than OPC.

Changing the chemical species to increase sulfate and alkali content causes some difference in the final expansion and the kinetics of expansion but affects little the presence or absence of 0.1% expansion.

Expansion when there is no exposure to high temperature differs from DEF expansion in that the expansion is very small.

Further, the distribution of sulfur in hardened specimens over time was observed. After DEF expansion, sulfur was found to migrate from the paste matrix to the cracks caused by DEF expansion, and to accumulate there. The formation of AFt guides associated with Ostwald ripening was found to be related to DEF expansion.

***Keywords**: DEF; maximum temperature at early age; alkali content; SO_3 content; expansion*

1. INTRODUCTION

Cracking due to delayed ettringite formation (DEF) is not only a problem for steam cured concrete products but can occur also in mass concrete where the concrete temperature exceeds 70°C as the internal temperature rises [1]. DEF cracking can occur when the three conditions of excessive SO_3 content, exposure to high temperature at young age, and sufficient water supply at service are simultaneously satisfied [2]. The revision of the Guidelines for Control of Cracking of Mass Concrete (Guidelines 2016) [3] includes the addition of clauses on DEF crack prevention. Even when these three conditions are met, there are cases where DEF cracking does not occur. Conditions under which expansion does not occur even when exposure of concrete to high temperature cannot be avoided have been studied under various material and mixing conditions. Much research on DEF expansion has been done, and it has been found that expansion/non-expansion areas can be distinguished by alkali of sodium equivalent (hereafter referred to as R_2O) and sulfate (SO_3) content in concrete, and various material conditions have been proposed [4]. R_2O content promotes the decomposition of ettringite at high temperature and SO_3 content is an important factor related to the initial amount of ettringite. These factors have been reported to be related to DEF expansion [4].

In previous studies, mainly R_2O content and SO_3 content were increased by addition to concrete as R_2SO_4. Thus far, little research has been done on the independent increase of R_2O and SO_3, and the discrete influence of each has remained unknown.

In this study, we investigated the influence of R_2O content and SO_3 content on DEF expansion using a mortar mix proportion in which R_2O and SO_3 were independently increased, assuming the mortar portion of concrete with unit cement content of 300 kg/m^3.

2. HANDLING OF DEF IN GUIDELINES FOR CONTROL OF CRACKING OF MASS CONCRETE (2016) JCI

The main purpose of revision of the guidelines (hereinafter referred to as guidelines) is to inspect and prevent the occurrence of DEF cracks. The guidelines prescribe the maximum allowable temperature based on the cement used and the R_2O content and SO_3 content in the concrete mixture (Guidelines 2016) [3].

Figure 2.1 shows the experimental results of the previously reported studies, and shows the relationship between final expansion and maximum temperature[1][4-13]. When concrete was exposed to high temperature of 70°C or more, DEF expansion of 0.1% or more can occur. Some cases show expansion of 0.1% or more even at 65°C. In the mortar bar test of alkali aggregate reaction (JIS A 1146: JIS A1146: Method of test for alkali-silica reactivity of aggregates by mortar-bar method), it is said that expansion of 0.1% or more is not harmless and that serious cracks will occur in concrete.

As the maximum temperature rises, the maximum value of the expansion also increases. The influence of the maximum temperature on DEF expansion is strong. When the maximum temperature is 70°C, some cases show 0.6% or more of expansion, but some cases have expansion of 0.1% or less. Even when the temperature exceeds 70°C, some cases have 0.1% or less of expansion.

Figure 2.2 shows the relationship between R_2O and SO_3 content and the presence or absence of expansion of 0.1% in the case of maximum temperature of 65°C to 70°C. Expansion of 0.1% or more was observed when R_2O content was 4 kg/m^3 or more and SO_3 content was 17 kg/m^3 or more. At the maximum temperature of 70°C, some cases reached expansion of 0.8%. When the maximum temperature was 65°C, maximum expansion dropped to 0.2%. When the R_2O content was 4 kg/m^3 or less or the SO_3 content was 17 kg/m^3 or less, expansion was 0.1% or less. The maximum temperature of concrete is based on 70°C, and when both R_2O content and SO_3 content are large, it is recommended that the maximum temperature limit be 65°C (Guidelines 2016) [3]. The pessimum effect has not been observed in research on DEF.

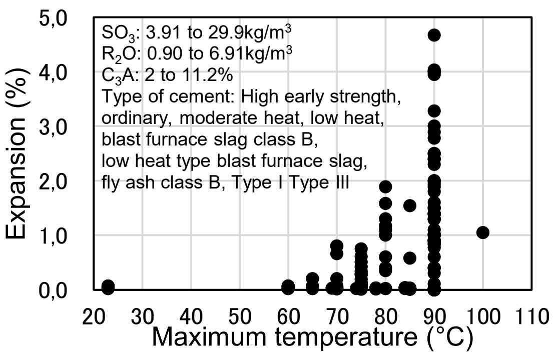


Figure 2.1: Relationship between maximum temperature in concrete and DEF expansion (Guidelines 2016) [3]

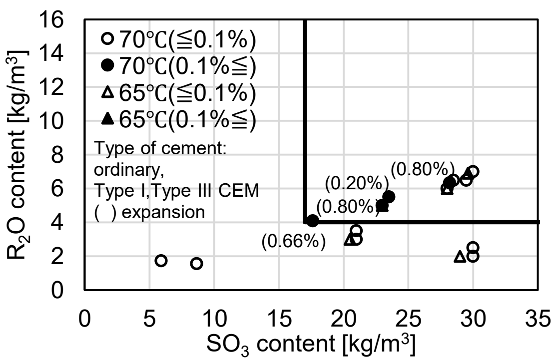


Figure 2.2: Influence of R_2O and SO_3 content in concrete on DEF expansion when maximum temperature: 70°C or under (Guidelines 2016) [3]

Figure 2.3 show the relationship between R_2O and SO_3 content and the presence or absence of 0.1% expansion for each maximum temperature among the data in Figure 2.1. Regardless of the type of cement, the range of R_2O affecting DEF cracking is 2 kg/m^3 to 7 kg/m^3 and the range of SO_3 is 4 kg/m^3 to 30 kg/m^3. Figure 2.3a shows the case where the maximum temperature of concrete is 65°C to 80°C. When R_2O content in the concrete was 3 kg/m^3 or less and SO_3 content was 9 kg/m^3 or less, the expansion was 0.1% or less in either case. Figure 2.3b shows the case where the maximum temperature exceeds 80°C and is below 90°C. When R_2O content was less than 3 kg/m^3 and SO_3 content was less than 9 kg/m^3, the expansion rate was less than 0.1% in most cements, but there was a case of expansion for high-early strength Portland cement. From this, the guidelines prescribe that the maximum temperature limit for high-early-strength Portland cement be 80°C.

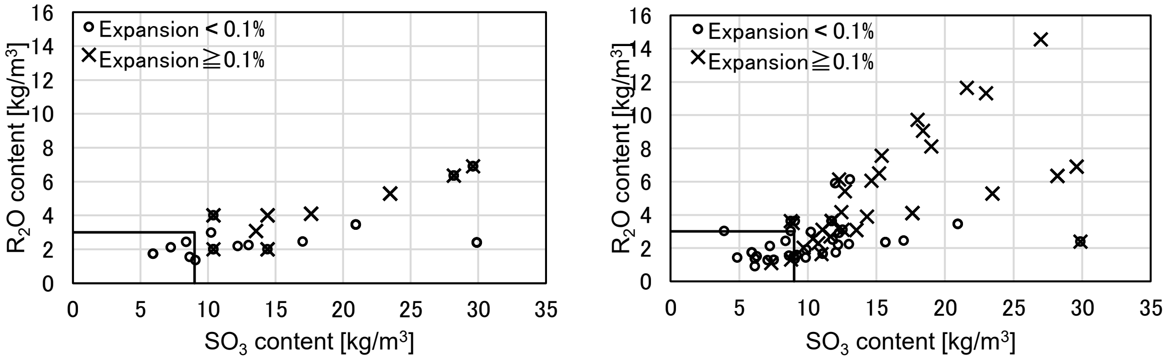


Figure 2.3: Influence of R_2O content and SO_3 content on DEF expansion (Guidelines 2016)[3]

As a summary of the above, Table 2.1 and Figure 2.4 show the threshold values of the maximum temperature when using high-early-strength (hereinafter referred to as HPC) and ordinary Portland cement (OPC). Basically, 70°C, at which temperature ettringite is decomposed, is the maximum temperature limit. However, when R_2O content in the concrete is 4 kg/m^3 or more and SO_3 content is 17 kg/m^3 or more, the maximum temperature limit is 65°C.

In addition, the threshold values of the maximum temperature of concrete can be 80°C when both R_2O content and SO_3 content in the concrete satisfy condition (1) and when the cement and admixture satisfy conditions (2) and (3) below.

(1) When HPC or OPC is used, make the R_2O content and SO_3 content in concrete 3.0 kg/m^3 or less and 9.0 kg/m^3 or less, respectively.

(2) Use moderate-heat or low-heat Portland cement.

(3) Portland blast-furnace slag cement B type (slag content ranging from 30% to 60%) or C type (slag content ranging from 60% to 70%), and Portland fly-ash cement type C (fly ash content ranging from 20% to 30%) are used. Ground granulated blast-furnace slag and fly ash are used at a mixing ratio equal to or higher than those of these cements. Silica fume for concrete is used at a mixing ratio of 10% or more.

Table 2.1: Threshold values of maximum temperature of concrete to prevent DEF cracking (Guidelines 2016) [3]

Cement	R_2O content and SO_3 content	Limit of maximum temperature
HPC & OPC	More than 4 kg/m^3 of R_2O and more than 17 kg/m^3 of SO_3	Less than 65°C
	More than 3 kg/m^3 of R_2O or more than 9 kg/m^3 of SO_3	Less than 70°C
	Less than 3 kg/m^3 of R_2O and less than 9 kg/m^3 of SO_3	Less than 80°C

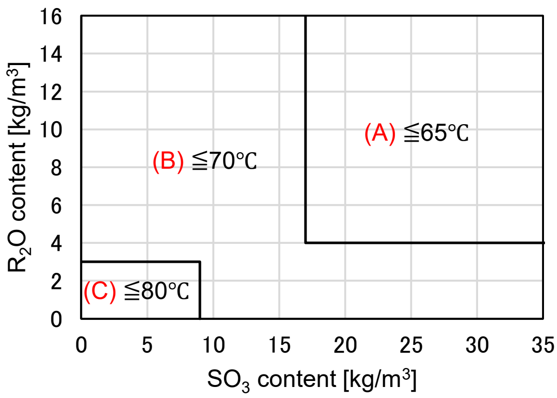


Figure 2.4: Threshold values of maximum temperature of concrete due to R_2O content and SO_3 content (Guidelines 2016)[3]

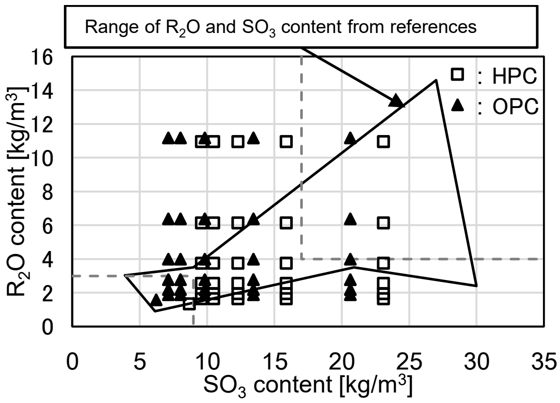


Figure 2.5: Content of R_2O and SO_3 in concrete in this study

3. EXPERIMENTAL METHOD

Cement was limited to two types of commercially available HPC (R_2O = 0.45%, SO_3 = 2.90%) and OPC (R_2O = 0.53%, SO_3 = 2.08%). The water cement ratio was 0.55 and the fine aggregate cement ratio was 2.5. For fine aggregate, crushed sand produced at Kurokawa in Morioka (saturated and surface-dry condition of 2.83 g/cm^3, fineness modulus of 2.89) was used in each experiment.

Figure 5 shows the levels of R_2O and SO_3 in each cement in this study. The closed broken line indicates the composition ranges of R_2O and SO_3 content of referenced previously published studies. Since the increment of R_2O and SO_3 was carried out by adding R_2SO_4, the referenced data ranges from the lower left to the upper right. Therefore, study of the influence of R_2O content and SO_3 content, respectively, is called for. In this experiment, R_2O content was increased with NaOH (sodium hydroxide) and SO_3 content was increased with $CaSO_4 \cdot 2H_2O$ (gypsum), respectively, and the investigation was carried out across a wider composition range than in previously reported studies.

Across four formulations of HPC, we changed the chemical species to KCl (potassium chloride) or $MgSO_4$ (magnesium sulfate) and compared them. The R_2O content and SO_3 content are the values converted in concrete, assuming the mortar portion of concrete with unit cement content of 300 kg/m^3.

In this experiment the maximum temperature was set to 70, 80 and 90°C. The heat treatment was to store the concrete at 20°C until 4 hours after mixing, then increase the heat to the maximum temperature at the rate of 20°C/h, maintain the maximum temperature for 12 h, then cool down the concrete at the rate of -20°C/h. After 24 hours, the specimens were demolded and stored at 20°C in a big water pool.

The length change test was conducted by the test method for length change of mortar and concrete described in JIS A 1129: Methods of measurement for length change of mortar and concrete—Part 3: Method with dial gauge, and measurement was performed every 2 weeks. Expansion judgment was based on the mortar bar test of alkali aggregate reaction, using the criteria that expansion of less than 0.1% is harmless, and expansion of 0.1% or more is harmful (Guidelines 2016 [3]). Long-term age is required for the occurrence of DEF, but in accelerated experiments, this period may be 180 days (Guidelines 2016)[3]. Here, the period of 200 days was used. The specimens were stored in the same big water pool mentioned previously.

4. RESULTS AND DISCUSSION

4.1 Effect of maximum temperature

Figure 6 shows the change in length of concrete with three different levels of R_2O and SO_3 content. The maximum temperatures used for HPC were 70, 80, and 90°C, and the maximum temperature for OPC was 90°C. The three levels of R_2O and SO_3 content of HPC were 1.7-9.6 kg/m^3 (R_2O = 1.7 kg/m^3, SO_3 = 9.6 kg/m^3), 6.2-15.9 kg/m^3, and 11.0-23.1 kg/m^3, and for OPC, the three levels were 1.9-7.1 kg/m^3, 6.4-13.4 kg/m^3, and 11.2-20.6 kg/m^3.

Hereinafter, the same description is applied for the type of cement and maximum temperature. When the R_2O content and SO_3 content were the same in HPC (Figures 4-1: a-c), the expansion increased as the maximum temperature rose. When the R_2O content and SO_3 content were 1.7-9.6 kg/m^3, DEF expansion of more than 0.1% was not observed at 70-90°C in this study. When the maximum temperature is 70°C or more, the risk of DEF expansion is higher the larger the R_2O content and SO_3 content. On the other hand, if R_2O content and SO_3 content are suppressed, there are cases where the maximum temperature does not expand to the range of 70-90°C. These results are similar to the results of previous reports.

In the actual concrete structure, the inside is exposed to high temperature due to the temperature rise caused by the hydration heat of the cement, and the risk of expansion and cracking due to DEF increases when the R_2O content and SO_3 content are large.

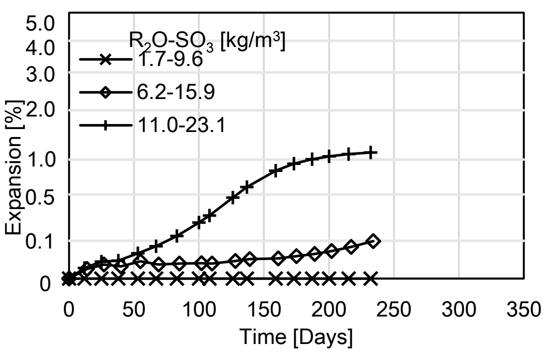
4.2 Effect of cement type

Comparing Figures 4.1:c, d, HPC expands more than OPC at the same maximum temperature, and the expansion speed is also much greater. When HPC is used, expansion rapidly increases from the beginning of expansion unlike for OPC, and expansion ends in a short time. The Y-axis represents the square root of expansion (%).

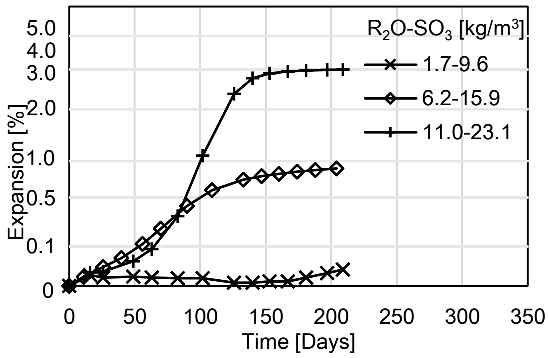
4.3 Effect of R_2O and SO_3 content

Figures 4.2a, 4.2b shows the relationship between R_2O content and expansion at 200 days and the relationship between SO_3 content and expansion at 200 days of HPC-90°C. As R_2O content and SO_3 content increase, the maximum value of expansion increases. For the monitoring duration considered, some concretes did not expand even when R_2O content and SO_3 content increased; however, for slow expansion cases, the absence of expansion might correspond to a latent period of DEF and expansion could occur for longer ageing durations. In Figure 4.2a, even when R_2O content was lowered to 3 kg/m^3 or less, many concretes expanded.

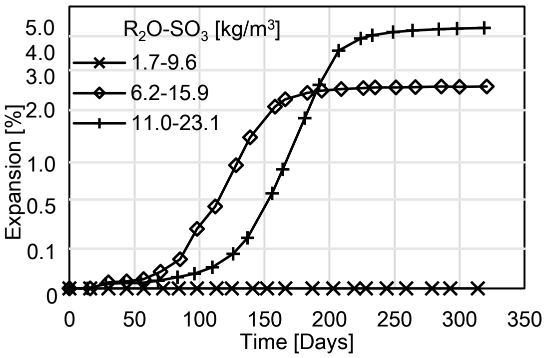
Since the R_2O content and SO_3 content each interact with DEF and the presence or absence of DEF expansion cannot be determined independently for each, we decided to study the combined effect of R_2O content and SO_3 content.



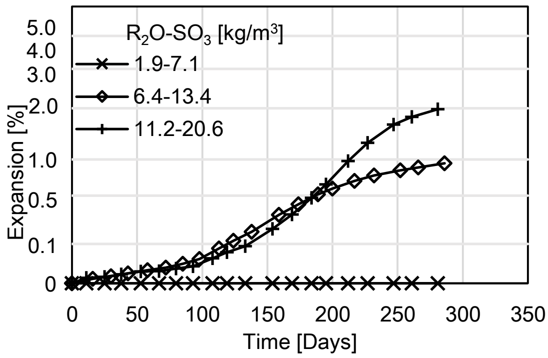
a. HPC-70°C



b. HPC-80°C



c. HPC-90°C



d. OPC-90°C

Figure 4.1: Expansion in HPC mortars cured at 70, 80, 90°C and OPC mortars cured at 90°C

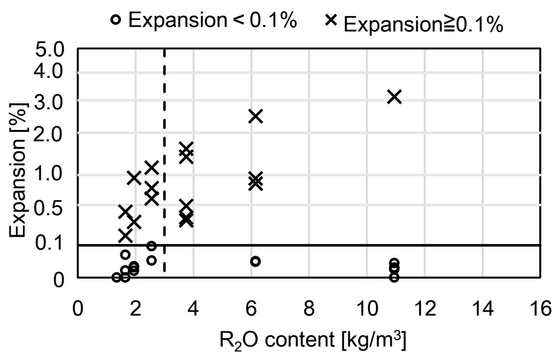


Figure 4.2a: Relationship between R_2O content and expansion (HPC-90°C, 200 days)

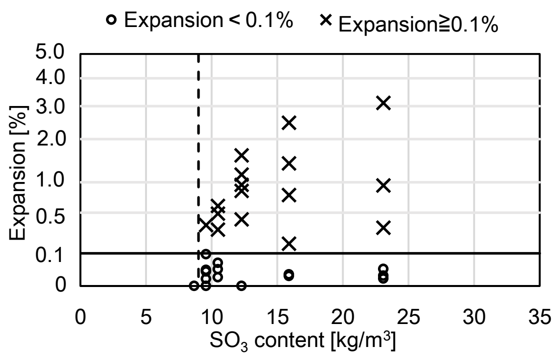
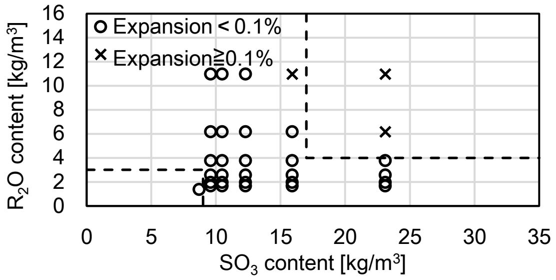


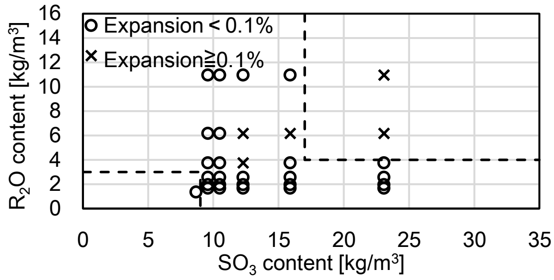
Figure 4.2b: Relationship between SO_3 content and expansion (HPC-90°C, 200 days)

4.4 Combined effect of maximum temperature, R2O content and SO3 content

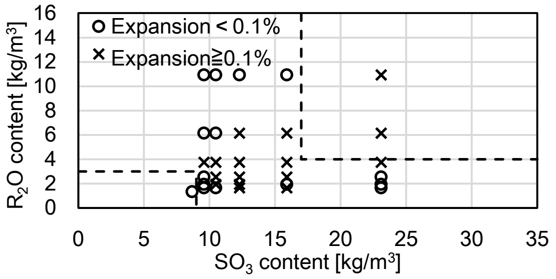
Figures 4.3 show the relationship between R_2O and SO_3 content in the concrete and the presence or absence of expansion for each cement and maximum temperature. Figures 4.3 a-c show this relationship at the maximum temperatures of 70°C, 80°C, and 90°C, respectively, for HPC. Figure 4.3 d shows the relationship at the lowest maximum temperature of 70°C for HPC, along with the relationships at 80°C and 90°C. Figure 4.3 e shows the relationship at the maximum temperature of 90°C for OPC. Figure 8f shows the relationship at the maximum temperature of 65°C or less indicated by the reference (JCI 2016). Figures 4.3 g, h show the combined results of the previous studies and the experimental results for the cases where the maximum temperature is over 65°C to 70°C, and over 70°C up to 80°C, respectively.



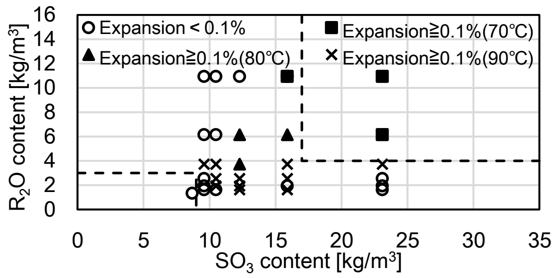
a. HPC-70°C, 200 days



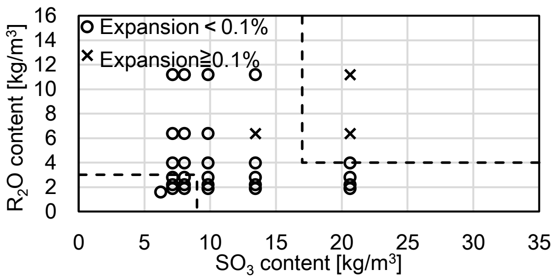
b. HPC-80°C, 200 days



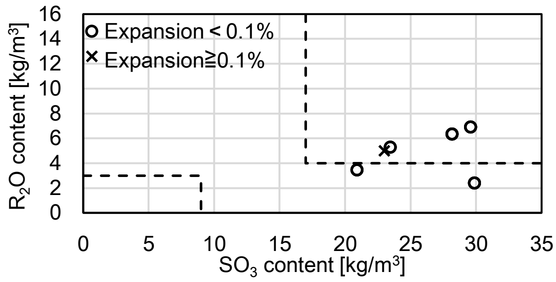
c. HPC-90°C, 200 days



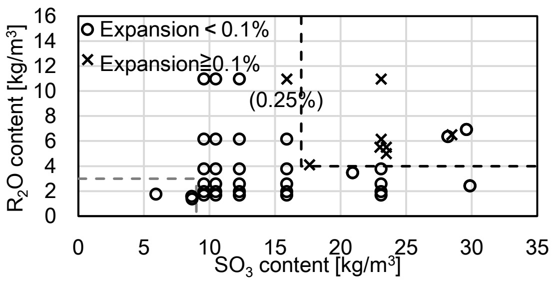
d. HPC-70, 80, 90°C, 200 days



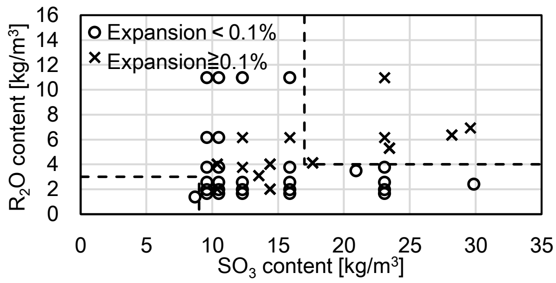
e. OPC-90°C, 200 days



f. Under 65°C (Guidelines 2016)



g. Over 65°C up to 70°C



h. Over 70°C up to 80°C

Figure 4.3: Relationship between R_2O and SO_3 content in concrete and expansion

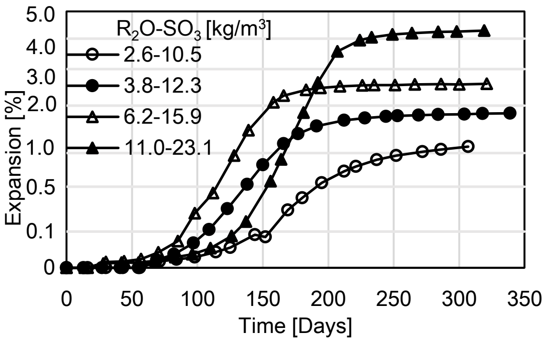
From Figures 4.3 a-c for HPC, as the maximum temperature rises, mortars with lower R_2O content and SO_3 content expand. Comparing Figures 8c and 8e, many of the HPC mortars expand even with less R_2O and SO_3 than OPC.

In Figure 4.3 d, when R_2O content is 3 kg/m^3 or more or SO_3 content is 12 kg/m^3 or more, expansion occurs even at 80°C, and when R_2O content is 6 kg/m^3 or more or SO_3 is 15 kg/m^3 or more, expansion also occurs at 70°C.

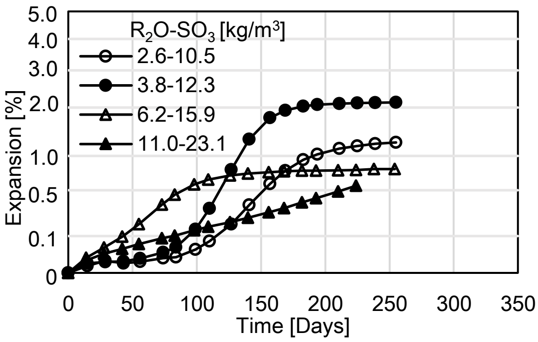
Figure 4.3 f is the data indicated by the guideline (Guidelines 2016)[3]. At temperatures over 65°C up to 70°C (Figure 4.3 g), even outside the range of expansion (R_2O content of 4 kg/m^3 or more and SO_3 content of 17 kg/m^3 or more) at the maximum temperature of 70°C, expansion of 0.25% was seen in the experimental results. In the case of over 70°C up to 80°C (Figure 8h), expansion did not occur when R_2O content was 3 kg/m^3 or less and SO_3 content was 9 kg/m^3 or less.

4.5 Effect of difference in chemical species

Figure 4.4 a shows the length change when R_2O and SO_3 are added as NaOH and $CaSO_4{\cdot}2H_2O$ for HPC-90°C, and Figure 4.4 b shows the length change when they are added as KCl and $MgSO_4$. Comparison at the age 200 days shows that expansion is almost the same for both chemical combinations at the level of R_2O and SO_3 of 2.6-10.5 kg/m^3 and 3.8-12.3 kg/m^3, respectively. When R_2O content and SO_3 content are 6.2-15.9 kg/m^3 and 11.0-23.1 kg/m^3, respectively, the expansion decreases by 1% or more when KCl and $MgSO_4$ are used. However, both levels result in expansion by 0.1% or more, and even if the chemical species changes, the influence on the evaluation of the presence or absence of expansion is small.



a. HPC-90°C, NaOH-$CaSO_4{\cdot}2H_2O$



b. HPC-90°C, KCl-$MgSO_4$

Figure 4.4: Influence of kind of chemicals added on DEF expansion of hardened specimens

4.6 Threshold values of the maximum temperature of concrete to prevent DEF cracking

In this experiment, expansion/non-expansion results for a wide range of R_2O and SO_3 content were obtained using two types of cement (HPC, OPC). In addition, the influence on the determination of the presence or absence of DEF expansion due to R_2O and SO_3 content was small even if the chemical species was changed. Figure 10 shows the range of the limit value corrected based on the results of this experiment. This supports the proposal of the threshold values of the maximum temperature of concrete to prevent DEF cracking indicated in the guidelines. Further, it was found that the threshold values of the SO_3 content need to be lowered to about 15 kg/m^3 from 17 kg/m^3 if the R_2O content is larger than 7 kg/m^3 in the JIS guidelines, as shown in Figure 4.5

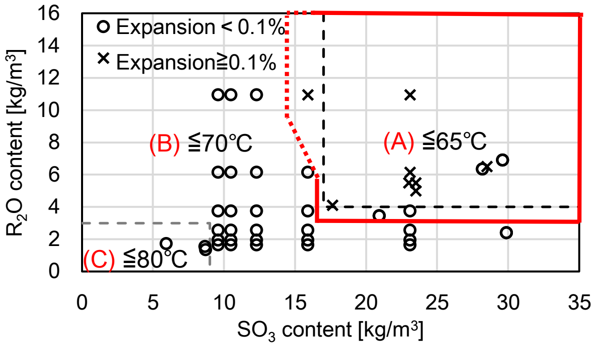
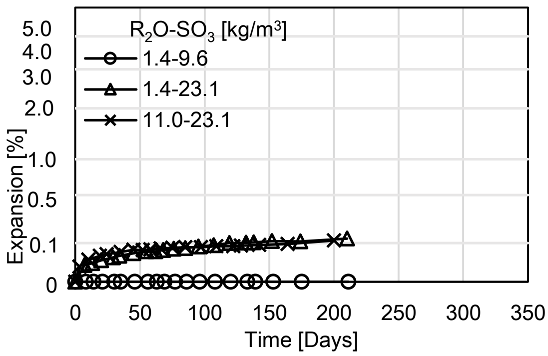


Figure 4.5: Range of R_2O content and SO_3 content where expansion can occur at each temperature (Over 65°C up to 70°C)

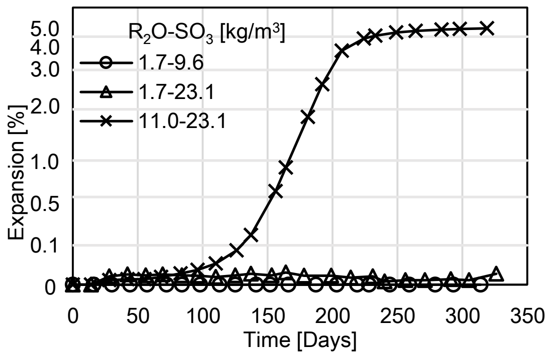
4.7 Effect of exposure to high temperature

Figure 4.6 a shows the length change when concrete is moist cured at 20°C, and Figure 4.6 b shows the length change when concrete is steam cured at 90°C. In both cases, HPC is used, and the chemical species are sodium hydroxide and gypsum. In the case of 20°C, expansion started at two levels, both with SO_3 content of 23.1 kg/m^3, at an early stage, but after 50 days of age, expansion ended, having reached about 0.1%. Converted into the content in cement, the SO_3 content is remarkably high at 7.7%. At these two levels, this can be regarded as sulfate attack at 20°C, due to the continuation of ettringite formation from the youngest age. This is a phenomenon completely different from DEF regenerated after decomposition of ettringite at early age.

DEF expansion was observed at SO_3 content of 23.1 kg/m^3 and R_2O content of 11.0 kg/m^3 at 90°C, and expansion exceeded 1% at 170 days of age. Expansion in the absence of exposure to high temperature differs from DEF expansion, and expansion is very small compared to when concrete is exposed to high temperature.



a. HPC-20°C



b. HPC-90°C

Figure 4.6: Expansion in HPC mortars cured at 20°C and 90°C

4.8 Distribution of sulfur in hardened specimens expanded by DEF

Figure 4.7 shows the BEI (back reflection electron image) of the polished surface of hardened specimens with DEF expansion (crushed sand 100%) and without DEF expansion (blast furnace slag fine sand (BF) 100%). Although the specimen was 150 days old and had been subjected to high-temperature curing of 100% crushed sand with DEF expansion, cracks with a width of 20-40 µm formed around the aggregate, connecting aggregate to aggregate. AFt was found to precipitate mainly in cracks. No cracks penetrating aggregate were observed. This structure is characteristic of those that have the characteristic structure of DEF expansion. The distribution of sulfur shows that high concentrations of sulfur are present in cracks and coincide with the distribution of AFt. On the other hand, AFt is detected in the XRD of the specimen with 100% BFS, but no agglomerated structure of AFt is observed. It can be seen that sulfur is uniformly distributed in the paste part. Regarding DEF cracking, there is a theory of crystal growth pressure due to AFt precipitation and a theory of paste expansion. According to the paste expansion theory, the decomposition of small AFt produced at early age is accelerated by the presence of CSH at high temperature, the desorbed sulfate ions are adsorbed by CSH, sulfate ions are released again by water supply during operation, and precipitation occurs near the CSH phase as small sized AFt. Along with this, the whole paste expands and cracks occur. It is thought that the cracks contained large sized AFt phase crystals due to residual sulfate ions, which were caused by Ostwald ripening, in which first small AFt precipitated and disappeared and then turned into large sized Aft phase. From these observations, it can be considered that in BF mortar that does not show expansion, fine AFt precipitates (regenerates) in the paste, but does not expand, and AFt does not undergo Ostwald growth. The mechanism by which paste expansion does not progress and DEF expansion does not occur when a large amount of BFS is used is still unclear.

5. CONCLUSIONS

We investigated the behavior of DEF expansion using two types of cement (HPC, OPC), across a wide range of experimental levels of R_2O and SO_3 content and chemical species, with exposure to high temperatures of 70°C, 80°C and 90°C.

- The results obtained here mostly agree with the contents of the JIS guidelines, thus supporting the threshold values of maximum temperature of concrete for the prevention of DEF cracking indicated by the guidelines. Regarding the range showing expansion at the maximum temperature of 70°C, when R_2O content is large, it is necessary to lower the threshold values of SO_3 content from about 17 kg/m^3 to about 15 kg/m^3.
- When only HPC is used and the maximum temperature is 70-90°C, expansion increases as the maximum temperature rises and the R_2O and SO_3 content increases. If R_2O content and SO_3 content are small, no expansion occurs.
- When only HPC is used and the maximum temperature is 90°C, the maximum value of expansion tends to increase as R_2O and SO_3 content increases as in previous studies.
- When comparing the two types of cement, HPC and OPC, expansion starts occurring from a lower content of R_2O and SO_3 for HPC.
- Changing the chemical species to increase sulfate and alkali content causes some difference in final expansion and in the kinetics of expansion but affects little the presence or absence of 0.1% expansion.
- Expansion when there is no exposure to high temperature differs from DEF expansion: expansion is very small compared with the case of exposure to high temperature.
- Observation of the distribution of sulfur in hardened specimens over time showed that after DEF expansion, sulfur migrates from the paste matrix to the cracks caused by DEF expansion and accumulates there. The formation of AFt guides associated with Ostwald ripening was found to be related to DEF expansion.

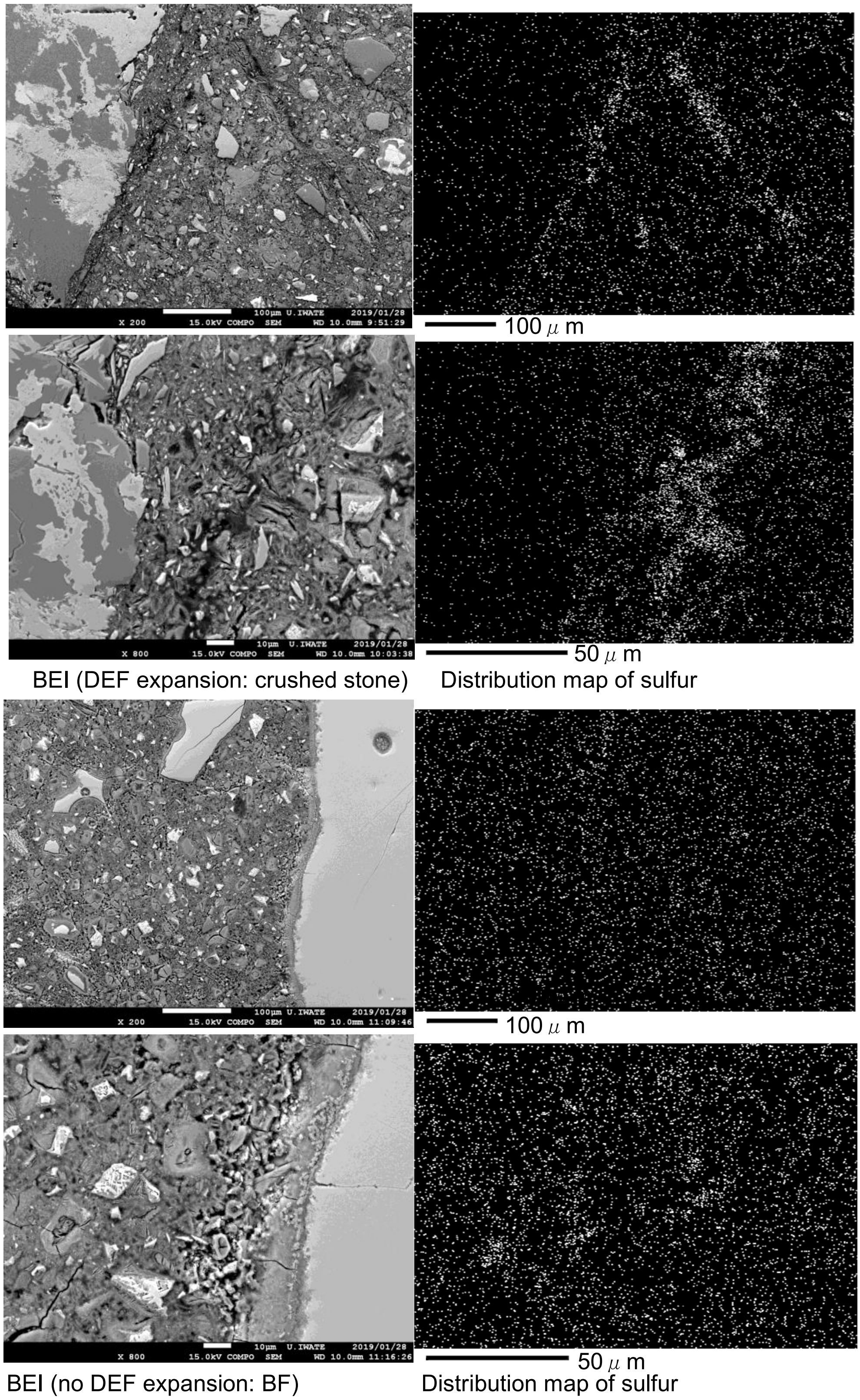


Figure 4.7: BEI (back reflection electron image) of polished surface of hardened specimens with DEF expansion (crushed sand 100%) and without DEF expansion (sand of BF 100%) (150 days)

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