

## EFFECTS OF PARTICLE SIZE, GRADING AND CONTENT OF REACTIVE AGGREGATE ON ASR EXPANSION OF MORTARS SUBJECTED TO AUTOCLAVE METHOD

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### ABSTRACT

Since an alkali-silica reaction (ASR) is intricately affected by different elements, it is very important to understand quantitatively the relationship between its expansion and these various factors. The characteristics of reactive aggregate were investigated from the viewpoint of these various factors.

In the present study, an autoclave method was selected to accelerate ASR in a mortar bar that was immersed in an NaOH solution and boiled inside an autoclave apparatus. The effects of the particle size, grading, and content of reactive aggregate on the ASR expansion of a mortar bar boiled in an NaOH solution inside the autoclave apparatus were examined. The parameters studied included: particle size, grading of reactive aggregate, the ratio of reactive aggregate to all aggregate by mass, and the aggregate-to-cement ratio (S/C).

The following results were obtained from the experiments:

- (1) The expansion of the mortar bar increased as the particle size or the F.M. of reactive aggregate decreased when the volume of reactive aggregate was the same.
- (2) There was a correlation between the expansion and the total surface area of reactive aggregate, regardless of the particle size and the grading of reactive aggregate when the volume of reactive aggregate was the same.
- (3) The expansion of the mortar bar was the same, regardless of the S/C when the grading of the reactive aggregate and the ratio of its volume of reactive aggregate to the alkali content ( $V/\text{Na}_2\text{O}$ ) of the mortar bar were equal.
- (4) The expansion of the mortar bar could be expressed by functions of the total surface area and the volume of reactive aggregate.

**Keywords:** Alkali-silica reaction, Particle size of reactive aggregate, Grading of reactive aggregate, Reactive aggregate content, Autoclave

### 1 INTRODUCTION

Alkali-silica reaction (ASR) progresses slowly over a long period and is a source of damage to structural members of concrete since it can seriously affect the durability and load resistance of the concrete structures. Therefore, the potential alkali-reactivity of aggregate must be identified in advance by an appropriately accelerated test.

On the other hand, ASR is intricately affected by such factors as the mix proportions of mortar or concrete, the materials, the characteristics of the reactive aggregate, alkali content, temperature, and humidity. Therefore, it is very important to

understand quantitatively the relationship between ASR expansion and such factors.

In the present study, the characteristics of reactive aggregate were investigated from various factors. Moreover, an autoclave method was selected to accelerate ASR in a mortar bar. In this autoclave method, a mortar bar was dried at 60°C for 24 hours to ease its permeability to the NaOH solution before being immersed into it and boiled inside an autoclave apparatus.

The present paper examines the effects of the particle size, the grading and content of reactive aggregate on ASR expansion of a mortar bar boiled in an NaOH solution inside an autoclave apparatus.

2 OUTLINE OF THE EXPERIMENT

2.1 Materials

The following materials were used in the present experiment: Cement: ordinary portland cement (0.68% Na<sub>2</sub>O equivalent, K<sub>2</sub>O/Na<sub>2</sub>O=1.9); Aggregate: non-reactive fine aggregate (NT) and reactive fine aggregates (T1). The reactive fine aggregate were bronzite andesite judged deleteriously reactive by the mortar bar method (JIS A 1146). Table 1 gives some of the properties of the aggregates used. Reagent grade NaOH was used for the preparation of the NaOH solution for immersion of the mortar bars.

2.2 Experimental Method [1]

Fig.1 shows a flow chart of the experimental method of the present study. The initial lengths of mortar bars were measured after demolding, and then the mortar bars were dried at 60°C for 24 hours to allow the NaOH solution to permeate them easily. The mortar bars were then immersed in NaOH solution with the given concentration inside a stainless steel container after drying and then boiled at 0.1 MPa for 72 hours in an autoclave apparatus. The changes in the length of the mortar bars were measured after they were taken out of the autoclave apparatus and moved to a constant temperature room (20°C) for cooling. While the mortar bars were in the constant temperature room for 24 hours, they were immersed in NaOH solution.

2.3 Test conditions

In all of the experiments, the autoclave treatment pressure and time were 0.1 MPa and 72 hours. The dimension of each mortar bar was 40×40×160 mm.

2.3.1 Experiment for the effects of the particle size and grading of reactive aggregate on ASR expansion of mortar bar

The ratios of water-to-cement (W/C) and aggregate-to-cement (S/C) of the mortar bars were kept constant at 0.5 and 2.25, respectively. The

aggregate used was sifted through several sieves (sieve apertures: 5, 2.5, 1.2, 0.6, 0.3 and 0.15 mm), and the aggregates that remained in each sieve (2.5, 1.2, 0.6, 0.3 and 0.15 mm) were used as “single size aggregates.” In addition, the remaining aggregates in the 1.2 and 0.15 mm sieves (hereafter called 2.5-1.2 and 0.3-0.15 mm, respectively) were mixed in ratios of 1:0, 0.85:0.15, 0.55:0.45, 0.3:0.70 and 0:1 by mass, respectively. Furthermore, the aggregates that remained in the 2.5, 1.2, 0.6, 0.3 and 0.15 mm sieves (hereafter called 5-2.5, 2.5-1.2, 1.2-0.6, 0.6-0.3 and

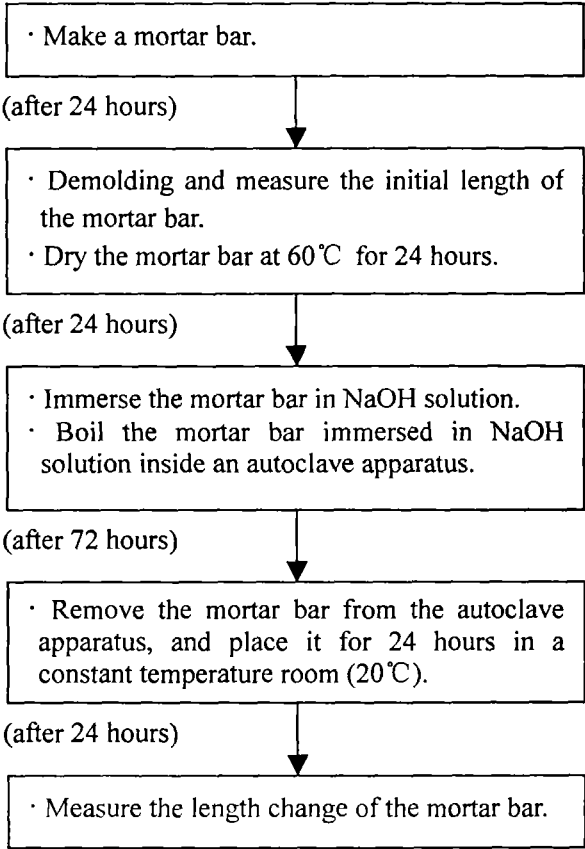


Fig.1 Flow chart of the experimental method

Table 1 Some properties of aggregates

Kinds of aggregate	SSD* (g/cm <sup>3</sup> )	Absorption (%)	Expansion at the age of six months by the mortar bar method (%) (JIS A 1146)
T1	2.60	1.93	0.281
NT	2.70	0.65	0.019

\*SSD: Saturated surface-dry particle density

0.3-0.15 mm) were mixed in the ratios shown in Table 2. Hereafter, the aggregate mixed with each particle size of aggregates is called the “mixed size aggregate.” The concentration of NaOH solution in which the mortar bars were immersed was set at three levels of 0.5, 1.0 and 2.0 mol/l.

### 2.3.1 Experiment for the effects of reactive aggregate content on ASR expansion of mortar bar

The aggregates that remained in the 2.5, 1.2, 0.6, 0.3 and 0.15 mm sieves were mixed together in ratios of 10:25:25:25:15 by mass, (according to JIS A 1146). The W/C of the mortar bars remained constant at 0.5. The S/C was set at four levels; 0.5, 0.8, 1.5 and 2.25. The ratio of reactive aggregate to all aggregate contained in the mortar by mass was set at four levels: 25, 50, 75 and 100%. The concentration of NaOH solution in which the mortar bars were immersed was kept constant at 1.0 mol/l.

## 3 RESULTS AND DISCUSSION

### 3.1 Effects of particle size and grading of reactive aggregate on ASR expansion of the mortar bar

Fig.2 shows the relationship between the expansion of mortar bars after the autoclave treatment and the particle size of the reactive aggregate. The mortar bars with a single particle size of reactive aggregate were immersed in 0.5, 1, and 2 mol/l NaOH solutions and treated at pressure of 0.10 MPa for 72 hours in the autoclave apparatus. This figure indicates that, for the mortar bars immersed in every concentration of NaOH solution, the expansion of the mortar bars increases as the particle size of the reactive aggregate decreases. Furthermore, for mortar bars with the reactive aggregate of every particle size, the expansion of mortar bars increases with the concentration of NaOH solution. In addition, as the particle size of reactive aggregate decreases, the rate of increase in the expansion with the concentration of NaOH solution becomes larger.

The reason is as follows. The total surface area of the reactive aggregate increases with the decrease in the particle size of the reactive aggregate for mortal bars having the same volume of reactive aggregate. Therefore, the amount of gel produced by ASR (hereafter ASR gel) increases as the particle size of the reactive aggregate decreases. Furthermore, ASR is accelerated as the concentration of NaOH solution, in which the mortar bars are immersed, increases. That is why the above tendencies appeared.

Fig.3 shows the relationship between the

Table 2 Mixing ratio of reactive aggregate of five particle sizes

①:	②:	③:	④:	⑤	F.M.
0.05:	0.35:	0.45:	0.10:	0.05	3.25
0.15:	0.25:	0.25:	0.25:	0.10	3.10
0.05:	0.05:	0.55:	0.20:	0.15	2.65
0.05:	0.05:	0.45:	0.15:	0.30	2.40

Particle size ①: 5-2.5 mm, ②: 2.5-1.2 mm  
③: 1.2-0.6 mm, ④: 0.6-0.3 mm  
⑤: 0.3-0.15 mm

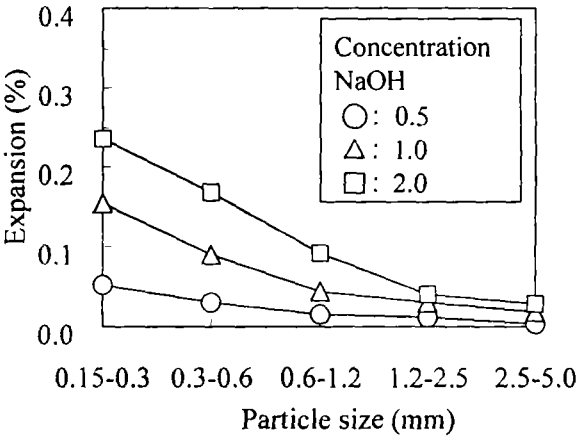


Fig.2 Relationship between expansion and particle size of reactive aggregate

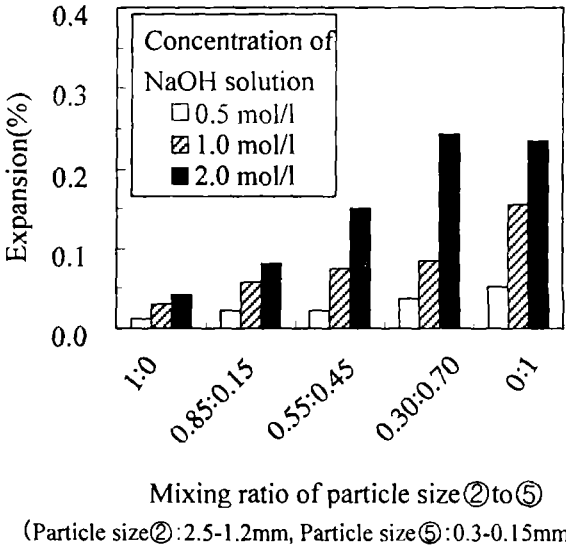


Fig.3 Relationship between expansion and mixing ratio of particle size ② to ⑤

expansion of mortar bars after autoclave treatment and the mixing ratio of the reactive aggregate of each particle size for the mortar bars which contain the reactive aggregates of the two kinds of particle size (2.5-1.2 mm and 0.3-0.15 mm). As shown in Fig.3, the expansion of the mortar bars immersed in every concentration of NaOH solution increases according to the content of the reactive aggregate of the smaller particle sizes of 0.3-0.15 mm. Furthermore, as the concentration of the NaOH solutions increases, the rate of increase in the expansion also becomes greater with increase in the content of reactive aggregate of 0.3-0.15 mm.

Fig.4 shows the relationship between the expansion of the mortar bars after autoclave treatment and the fineness modulus (F.M.) of reactive aggregate. In this case, the reactive aggregate of five particle sizes (2.5-1.2, 1.2-0.6, 0.6-0.3, 0.3-0.15 mm) was mixed and used for the production of mortar. This figure indicates that for the mortar bars immersed in 1.0 and 2.0 mol/l NaOH solutions, the expansion of the mortars increases as the F.M. decreases.

From the above results, the expansion of the mortar bars after autoclave treatment increases as the particle size or the F.M. of reactive aggregate decreases. The reason is as follows. When the particle size or the F.M. of reactive aggregate is smaller, a large amount of ASR gel is produced because the total surface area of the reactive aggregate in the mortar bar increases. As a result, the expansion of the mortar bar increases as the particle size or the F.M. of the reactive aggregate becomes smaller. The above shows that the total surface area of the reactive aggregate is an important factor affecting ASR expansion. Therefore, we examine the effect of the total surface area of the reactive aggregate on the ASR expansion of the mortar bars. In this experiment, the total surface area was calculated as follows.

The length, breadth and thickness of the aggregate that remained in each sieve (sieve apertures: 2.5, 1.2, 0.6, 0.3 and 0.15 mm) were measured. The particle size of aggregate was then derived from Eq. (1):

$$\frac{d}{D} = 0.98 \left( \frac{2nm^2}{m^2 + 1} \right)^{\frac{1}{2}} \tag{1}$$

$$m=B/T, \quad n=L/B$$

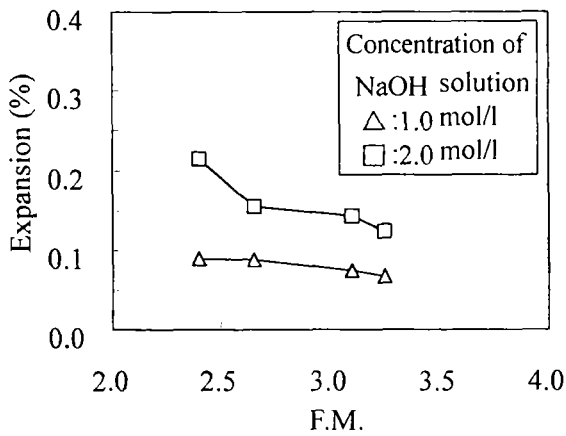


Fig.4 Relationship between expansion and fineness modulus (F.M.)

where d is the diameter of a circle having the same area as the projection of the particle (the projected area diameter) when viewed in its most stable position; D is the sieve aperture; and L, B, and T are the length, breadth, and thickness of the particle.

This equation, proposed by Heywood [2], calculates the diameter of a circle having the same area as the projection of a particle when viewed in its most stable position (i.e. the projected area diameter: d). The projected area diameter (d) derived from Eq. (1) is related to the surface area of the particle. If the particle is convex and arranged randomly, the surface area (s) of the particle is four times as large as the projected area (A) of the particle, regardless of the shape of the particle ( $s=4A$ ) [3]. That is to say, the surface area of the particle is equal to a sphere having diameter d. Accordingly, in the present study, the projected area diameter (d) derived from Eq. (1) was used to represent the particle size of the aggregate. The total surface area (S) of the aggregate contained in a mortar bar was estimated using the projected area diameter (d) as follows. The surface area (s) and volume (v) of an aggregate are  $s=\pi d^2$  and  $v=\pi d^3/6$ , respectively. The number (n) of aggregates contained in a mortar bar is given by the equation  $n=M/(\rho v)$  where the mass and density of the aggregates contained in a mortar bar are M and  $\rho$ , respectively. The total surface area (S) of aggregates contained in a mortar bar is given by the equation  $S=sM/(\rho v)$ . Therefore, the total surface area (S) is derived from Eq. (2).

$$S = \frac{6M}{\rho d} \tag{2}$$

Fig.5 shows the relationship between the ASR expansion of mortar bars and the total surface area of the reactive aggregate contained in them. It also shows the results for the mortar bars immersed in 1.0 and 2.0 mol/l NaOH solution. This figure shows that when the concentration of NaOH solution in which the mortar bars were immersed is the same, the expansion of mortar bars increases linearly with the total surface area of the reactive aggregate contained in the mortar bar. Single size or mixed size of reactive aggregate is not relevant. Furthermore, there is a correlation between the expansion and the total surface area of the reactive aggregate. Therefore, the effect of the particle size and grading of the reactive aggregate on ASR expansion of the mortar bar immersed in NaOH solution can be explained according to the size of the total surface area of the reactive aggregate contained in a mortar bar.

### 3.2 Effect of the reactive aggregate content on ASR expansion of mortar bar

Fig.6 shows the relationship between the expansion of mortar bar and the ratio of reactive aggregate to all aggregate contained in mortar bar by mass (hereafter the mixing ratio of reactive aggregate) when the aggregate-to-cement ratio (S/C) of a mortar bar was 0.5, 0.8, 1.5 and 2.25. In this case, the mortar bars were immersed in a 1 mol/l NaOH solution and boiled in the autoclave apparatus. This figure indicates that the expansion of the mortar bars after autoclave treatment increased with the mixing ratio of reactive aggregate when the S/C of the mortar bars is 0.5. This figure also shows the tendency that, for mortar bars with S/C=0.8, the expansion of mortar bars increases with the mixing ratio of reactive aggregate up to 75%, even though the rate of increase of expansion is smaller than the mortar bars with S/C=0.5. However, the expansion of the mortar bars decreases as the mixing ratio of reactive aggregate increases when the mixing ratio of reactive aggregate is larger than 75%. On the other hand, the expansion of mortar bar decreases as the mixing ratio of the reactive aggregate increases when the S/C of the mortar bar was 1.5 and 2.25. These phenomena could be explained as follows [4]: ASR expansion increases with reactive aggregate content because the amount of ASR gel produced by ASR increases with the reactive aggregate content. However, when the reactive aggregate content in a certain fixed amount of alkali increases excessively,  $\text{Na}^+$  and  $\text{K}^+$  which act on the reactive mineral deconcentrates and the amount of  $\text{Na}^+$  and  $\text{K}^+$  which contacts to surface of the reactive aggregate

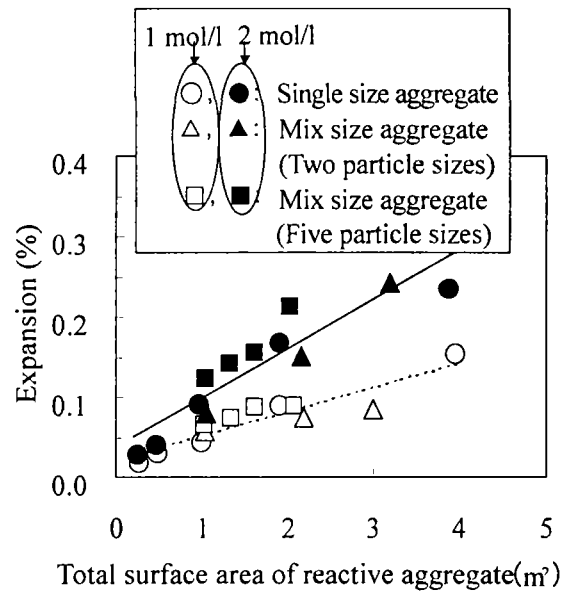


Fig.5 Relationship between expansion and total surface area of reactive aggregate

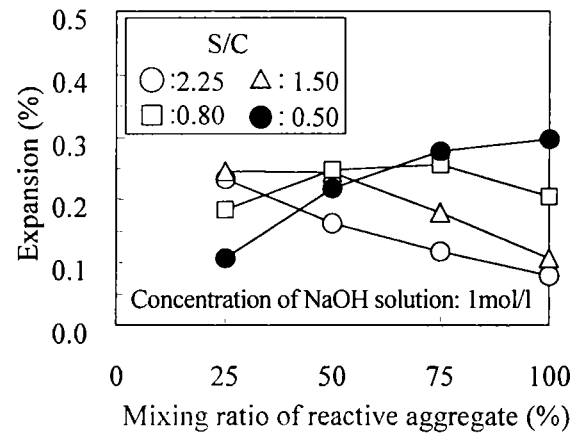


Fig.6 Relationship between expansion and mixing ratio of reactive aggregate

decreases relatively because the reactive aggregates approach each other, contracting the distance. As a result, ASR expansion slows down because ASR cannot proceed actively and the amount of ASR gel decreases. For this reason, it is considered that in the mortar bar of S/C=2.25 and 1.5, which has a large amount of the aggregate, the expansion decreases as the mixing ratio of the reactive aggregate increases.

The above result suggests that ASR expansion of the mortar bar is affected by the volume of the reactive aggregate. Therefore, we examined the relationship between ASR expansion of the mortar bar and the volume of the reactive aggregate. The

alkali content contained in mortar changes with the S/C of mortar because the quantity of NaOH solution that permeates the mortar bar and the cement content of the mortar bar change according to the S/C of the mortar bar. Therefore, we consider the ratio of the volume of the reactive aggregate to the alkali content contained in mortar (hereafter  $V/\text{Na}_2\text{O}$ ) and examine the relationship between ASR expansion and  $V/\text{Na}_2\text{O}$ . In this case, the alkali content contained in a mortar bar was estimated as follows. The quantity of the NaOH solution that permeated a mortar bar was measured. The amount of NaOH was calculated using the quantity of NaOH solution. The amount of NaOH was converted into the amount of  $\text{Na}_2\text{O}$ . The alkali content of mortar was calculated as the sum of  $\text{Na}_2\text{O}$  that permeated mortar and  $\text{Na}_2\text{O}$  from the cement in mortar.

Fig.7 shows the relationship between the expansion of the mortar bar and  $V/\text{Na}_2\text{O}$ . This figure indicates that the ASR expansion of the mortar bar increases as the volume of the reactive aggregate increases ( $V/\text{Na}_2\text{O}$  becomes larger), regardless of the S/C of the mortar bar. However, the ASR expansion decreases as the volume of reactive aggregate increases when  $V/\text{Na}_2\text{O}$  is greater than a certain value. The expansion of mortar bar is the same when the  $V/\text{Na}_2\text{O}$  of mortar bar is the same, regardless of the S/C of the mortar bar and the mixing ratio of the reactive aggregate. Furthermore,  $V/\text{Na}_2\text{O}$  that maximizes the expansion of the mortar bar is nearly the same, regardless of the S/C of the mortar bar. In the reactive aggregate used for this experiment the expansion of mortar bar maximizes when  $V/\text{Na}_2\text{O}$  is about 11. This value is the pessimum for the  $V/\text{Na}_2\text{O}$ . The pessimum value for  $V/\text{Na}_2\text{O}$  might exist because ASR expansion increases with the reactive aggregate content and because the amount of ASR gel increases with the reactive aggregate content. However, ASR cannot proceed actively when the reactive aggregate content increases excessively in a certain fixed amount of alkali because the amount of  $\text{Na}^+$  and  $\text{K}^+$  that acts on the reactive mineral decreases relatively as stated previously. As a result, the pessimum value for  $V/\text{Na}_2\text{O}$  appeared.

### 3.3 Effects of total surface area and volume of reactive aggregate on ASR expansion of the mortar bar

In Fig.7, the total surface area of reactive aggregates are the same when the volume of reactive aggregates are the same because the grading of the aggregates contained in every mortal bar are the same. Therefore, it cannot be known to what degree

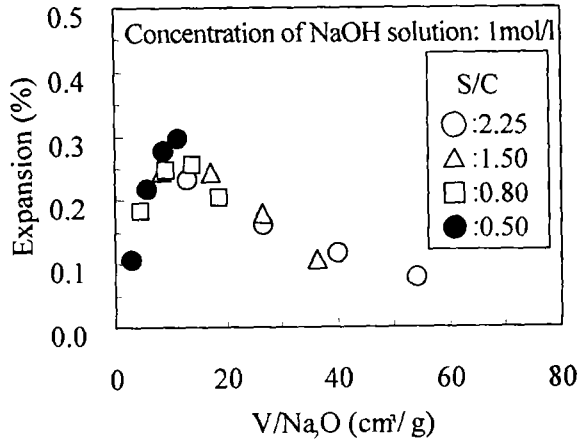


Fig.7 Relationship between expansion and  $V/\text{Na}_2\text{O}$

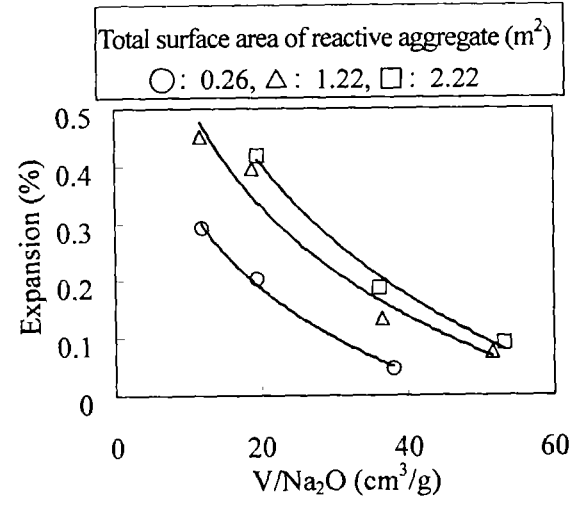


Fig.8 Relationship between expansion and  $V/\text{Na}_2\text{O}$

the total surface area and the volume of reactive aggregate affect the expansion of the mortar bar as shown in Fig.7. Consequently, we examine the relationship between ASR expansion and  $V/\text{Na}_2\text{O}$  for the mortar bar with the same total surface area of reactive aggregate. In this case, the grading of reactive aggregate and S/C of mortar bar are changed variously.

Fig.8 shows the relationship between the expansion and the  $V/\text{Na}_2\text{O}$  of the mortar bars when the total surface area of reactive aggregate contained in them are 0.26, 1.22 and 2.22  $\text{m}^2$ , respectively. Fig.8 indicates that the expansion of the mortar bar decreases as  $V/\text{Na}_2\text{O}$  increases, regardless of the total surface area of the reactive aggregate. Moreover, the expansion of the mortar bar increases as the total surface area increases when the  $V/\text{Na}_2\text{O}$  of the

mortar bar is the same. Furthermore, there was high correlation when the relationship between the expansion and  $V/Na_2O$  was approached by a logarithmic equation (Eqs.(3), (4), and (5)) for each mortar bar with the same total surface area of reactive aggregate:

$$S=0.26\text{ m}^2: E_x = -0.213 \ln \left( \frac{V}{Na_2O} \right) + 0.825 \quad (3)$$

$$(r = 0.998)$$

$$S=1.22\text{ m}^2: E_x = -0.277 \ln \left( \frac{V}{Na_2O} \right) + 1.160 \quad (4)$$

$$(r = 0.981)$$

$$S=2.22\text{ m}^2: E_x = -0.332 \ln \left( \frac{V}{Na_2O} \right) + 1.339 \quad (5)$$

$$(r = 0.994)$$

where  $E_x$ : expansion of mortar bar after autoclave treatment,  $S$ : total surface area of reactive aggregate,  $r$ : coefficient of correlation.

Now, we focus our attention on the coefficient (a) and intercept (b) of the above three regression equations. It is considered that coefficient (a) and intercept (b) change with the total surface area of the reactive aggregate because a different factor for the mortar bar with the same  $V/Na_2O$  is only the total surface area of the reactive aggregate. The relationship between the total surface area of reactive aggregate and coefficient (a) or intercept (b) is shown in Figs.9 and 10. Fig.9 shows a negative correlation between the coefficient (a) of the logarithmic equation and the total surface area of the reactive aggregate; both relations can be expressed in an approximate expression shown in Eq.(6). Furthermore, Fig.10 shows high correlation between the intercept (b) of the logarithmic equation and the total surface area of reactive aggregate; both relations can be expressed in an approximate expression as shown in Eq.(7). Eq.(8) can be obtained from Eqs.(3) to (7).

$$a = -0.0607 S - 0.199 \quad (r = 0.998) \quad (6)$$

$$b = 0.293 S + 0.767 \quad (r = 0.994) \quad (7)$$

Fig.11 indicates the comparison between the measured value shown in Fig.8 and the calculated

value for the expansion of the mortar bar. This figure shows that the calculated value from Eq.(8) approximately agrees with the measured value. Therefore, the expansion of the mortar bars can be

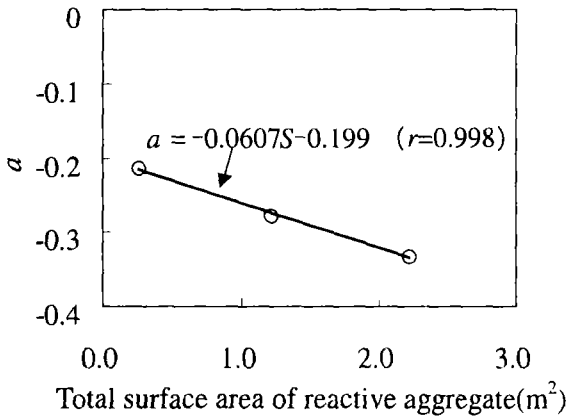


Fig.9 Relationship between coefficient (a) and total surface area of reactive aggregate

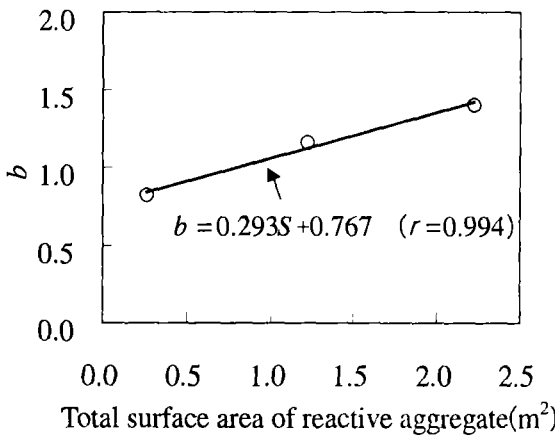


Fig.10 Relationship between intercept (b) and total surface area of reactive aggregate

expressed by functions of the total surface area and the volume of the reactive aggregate.

$$E_x = (-0.0607 S - 0.199) \ln \left( \frac{V}{Na_2O} \right) + 0.293 S + 0.767 \quad (8)$$

In the above discussion, we could not examine mortar bars whose  $V/Na_2O$  is smaller than 11 (that is, the region in which the expansion of the mortar bar increases with  $V/Na_2O$  as shown in Fig.7.). However,

for a mortar bar whose  $V/Na_2O$  is smaller than 11, the expansion of the mortar bar can be expressed by functions of the total surface area and the volume of the reactive aggregate. In the future we will examine a mortar bar whose  $V/Na_2O$  is smaller than 11.

## 4 CONCLUSIONS

In the present study, an accelerated test in which mortar bars immersed in NaOH solution were boiled in an autoclave apparatus was conducted. We examined the effects of the particle size, grading and content of the reactive aggregate on ASR expansion of mortar bars boiled in NaOH solution. The following results were obtained:

- (1) The expansion of mortar increased as the particle size or F.M. of reactive aggregate decreases when the volume of reactive aggregate was the same.
- (2) There was a correlation between the expansion and the total surface area of reactive aggregate, regardless of the particle size and grading of the reactive aggregate when its volume was the same.
- (3) The expansion of the mortar bars was the same, regardless of the S/C, when the grading of reactive aggregate and  $V/Na_2O$  of the mortar bar were the same, respectively.
- (4) It was seen that the expansion of mortar bars can be expressed by functions of the total surface area and the volume of reactive aggregate.

## ACKNOWLEDGEMENT

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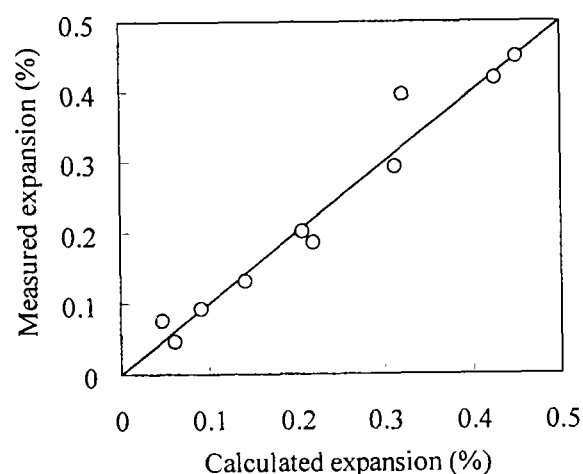


Fig.11 Comparison between measured expansion and calculated expansion

## REFERENCES

- [1] T. Kuroda, S. Inoue, A. Yoshino and S. Nishibayashi., ASR Expansion Characteristics of Mortar bars Immersed in NaOH Solution by Autoclave Method, Proc. of the 11th International Conference on Alkali-Aggregate Reaction, Quebec, Canada, 2000, pp.375-384.
- [2] J. K. Beddow, Particle Science and Technology, Chemical Publishing Co., Inc., 1980, pp.413-428.
- [3] S. Miwa, Powder Technology, Asakurashoten, Japan, 1972, pp.41-64.
- [4] W. Koyanagi, et al., Alkali-Aggregate Reaction in Concrete Structures, Rikogakusya, Japan, 1990, pp.25-29.