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STUDY ON THE EVALUATION OF ALKALI REACTIVITY IN AGGREGATE - TESTING CONDITIONS WITH THE MORTAR BAR METHOD -

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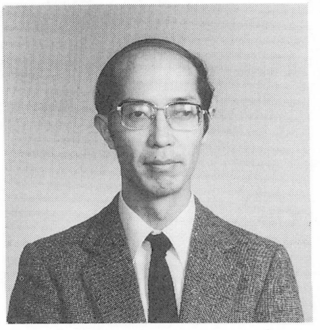

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SYNOPSIS

In order to prevent concrete damage from alkali aggregate reaction, it is important to know the potential reactivity of aggregate in advance and to take the necessary precautioning measures. ASTM chemical and mortar bar methods have been adopted widely throughout the world for determining the potential reactivity of aggregates. However, before this mortar bar method is effectively adopted in Japan, there are many factors that must be investigated, such as the mix proportion of the mortar and several other testing conditions. Consequently, this study was initiated to clarify the effects of various factors on the expansion due to alkali aggregate reaction and to develop a useful mortar bar method for determining the degree of alkali aggregate reaction in Japan.

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1. INTRODUCTION

In order to prevent cracking damage to concrete structures due to alkali aggregate reaction, it is important to develop a test method for evaluating the reactivity of aggregate. In Japan, ASTM chemical and the mortar bar method have been tentatively adopted for determining the reactivity of aggregate. With these test methods, however, we can only determine the potential alkali reactivity of the aggregate itself. The results obtained by these test methods do not always give direct information on whether the deleterious reaction is taking place in the concrete containing the aggregate. There are still some problems when the results obtained by these methods are used for estimating the expansion and the cracking damage of concrete.

Although there are some problems, as described above, there is no doubt that these test methods are very effective in determining whether the potentially reactive aggregate actually causes expansion under the presence of cement alkali.

These test methods were developed in the U.S.A., and there are some problems in applying these methods directly to aggregate in Japan. To improve the effectiveness and practicality of the mortar bar method, by which the reactivity of aggregate can be estimated directly from the expansion, it is necessary to investigate in detail the testing conditions and primary factors which affect the results of testing.

In this study, the effects of several factors, such as the water-cement ratio, the compacting conditions, and immersion into alkali solution, were investigated and discussed. At the same time, the changes in the mechanical properties of mortar due to alkali aggregate reaction were studied.

2. EXPERIMENTS

2.1 Materials

(1) Aggregate
Four kinds of reactive aggregate which were reported to have developed cracking damage in actual concrete structures (O, T1, T2, Q), and one non-reactive aggregate (N) were used. The physical and chemical properties of these aggregates are shown in Table 1. and in Fig. 1. The results of mortar bar tests using the reactive aggregates are shown in Fig. 2. Non-reactive river sand (Sendai river, Tottori) was used in a part of the experiment.

Table 1 Physical and chemical testing results of aggregates

Symbols of aggregate	Specific gravity	Water absorption (%)	ASTM chemical method (m mol/l) Sc	Rc	Sc/Rc
O	2.25	1.81	732	177	4.14
T1	2.60	1.93	558	101	5.53
T2	2.64	1.48	301	68	4.43
Q	2.56	1.67	384	93	4.13
N	2.70	0.65	30	22	1.36
Ø	2.67	0.28	114	38	3.00

(2) Cement
Normal portland cement samples having an alkali content of 0.50, 0.63, 0.80, and 1.00% as Na_2O equivalent were used.

(3) Added alkali and alkali solution for immersion
NaOH was used in most cases, and NaCl was used in the other cases, to control the alkali content of the mortar. 10% solutions of NaCl,

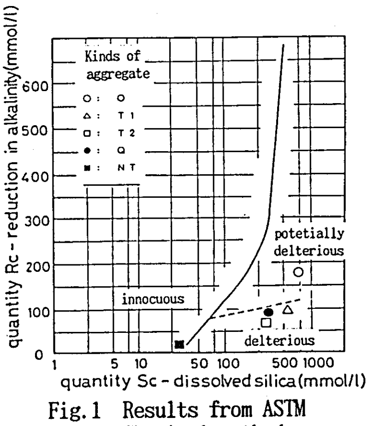

Fig. 1 Results from ASTM Chemical method

NaOH, and KOH were used to immerse the mortar specimens.

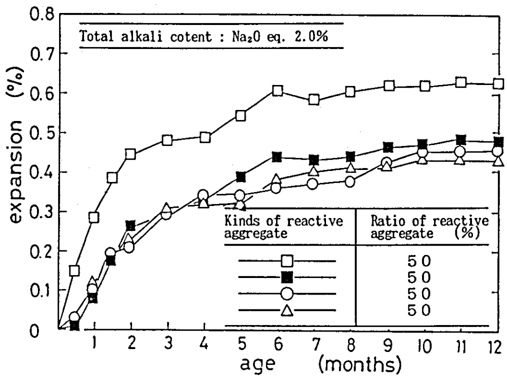

Fig. 2 Results from ASTM mortar bar method

2.2 Experiment procedures

In most cases, the mortar was proportioned so as to have a water-cement ratio of 0.45 and a aggregate-cement ratio of 2.25. The mortar bar specimens were produced according to JIS R 5201. The dimensions of the specimens were 4x4x16cm (JIS specimens) and 2.54x2.54 x28.5cm (ASTM specimens). The mortar specimens were cast and demolded after being kept in the mold for 24 hours in a thermostatic chamber at 20°C. After the initial lengths were measured, the specimens were stored in several conditions. After being stored for 24 hours in a thermostatic chamber at 20°C, the expansion of the specimens was measured. Except with the alkali solution immersion, the storage conditions of the specimens were set according to the ASTM mortar bar method for 38°C. The specimens at 20°C were placed just over the water surface in a closed vessel in the thermostatic chamber.

Table 2 Testing plan and conditions

Materials	Aggregate	Reactive	O, T1, T2
		Non reactive	N
	Ratio of reactive agg. (%)		50
	Cement	0.5, 0.8, 1.0 (Na_2O eq. (%))	
	Alkali	NaOH	
Testing conditions	Total alkali content Na_2O eq. (%)	Cement only (0.5, 0.8, 1.0) 1.0, 1.5, 2.0 (Cement (0.5(%)+ NaOH))	
	Mix proportions	W/C	0.45, 0.50, 0.55
		S/C	2.25
	Dimension of specimen (cm)		4×4×16, 2.54×2.54×28.5

Table 3 Testing plan and conditions (Compacting time)

Materials	Aggregate	Reactive	O
		Non reactive	N
	Ratio of reactive agg. (%)		50
	Cement	0.5 (Na_2O eq. (%))	
	Alkali	NaOH	
Testing conditions	Total alkali content	Cement only (0.5) 2.0 (Na_2O eq. (%))	
	Mix proportions	W/C	0.45
		S/C	2.25
	Dimension of specimen		4×4×16 cm
	Compacting time (sec.)		First layer : 0, 30, 60, 120 Second layer : 60, 120

2.3 Testing plan

(1) Water-cement ratio and dimensions of the specimens

Three water-cement ratios, 0.45, 0.50, and 0.55, were selected. The sand-cement ratio was 2.25. Expansion of each mortar specimen was measured over time. Two kinds of specimens were adopted, ASTM and JIS. The effects of the dimensions of the specimens on expansion were examined. The Test plan for these parameters is shown in Table 2.

(2) Compacting time

The effects of the compacting time of the mortar on the expansion of mortar specimens were examined. The compacting time using a vibration table after compacting with a rod were 0 (rod only), 30, 60, and 120 seconds. Compacting was made in one and two mortar layers. In the two layer specimens, each layer was compacted for half of the total compacting time. For the rod only specimens, the mortar was casted in two layers, and each layer was compacted 15 times with a rod. The test plan is shown in Table 3.

(3) Immersion into alkali solution
A 10% solution each of NaCl, NaOH and KOH, sea water, and water were used for immersing specimens. The total alkali content of the mortar was adjusted to 0.5% (cement only) and 2.0% as Na_2O equivalent, and the penetration of alkali from outside was investigated. At the same time, to examine the effect of pre-curing, some mortar specimens were immersed into an alkali solution after curing for 28 days in water at 20°C, and the expansion of these specimens was compared with that of specimens having no pre-curing. Each alkali solution was at 20 ± 2 °C and was renewed every three months. The test plan is shown in Table 4.

Table 4 Testing plan and conditions (Liquids for immersion)

Materials	Aggregate	Reactive	O, T1, T2, Q
		Non reactive	N
	Ratio of reactive agg. (%)		50
	Cement	0.5 (Na_2O eq. (%))	
	Alkali	NaOH	
Testing conditions	Total alkali content	Cement only (0.5) 2.0 (Na_2O eq. (%))	
	Mix proportions	W/C	0.45
		S/C	2.25
	Dimension of specimen	4 × 4 × 16 cm	
	Curing condition	No pre-curing, Pre-curing in water for 28 days	
	Liquids for immersion (density %)	Water, Sea water (3.6), NaOH (10), NaCl (10), KOH (10)	

(4) Strength and Young's modulus
The dynamic Young's modulus and the modulus of rupture and compressive strength (1, 3, and 6 months) were measured, and these values were compared with the expansion of the specimens. In this experiment, NaCl was used for the additional alkali, because too much NaOH causes hardening of fresh mortar and makes it difficult to cast the mortar, which results in lower strength of mortar. The test plan is shown in Table 5.

Table 5 Testing plan and conditions (Relationship between expansion and mechanical properties of mortar)

Materials	Aggregate	Reactive	O, T1
		Non reactive	O
	Ratio of reactive agg. (%)		50
	Cement	0.63 (Na_2O eq. (%))	
	Alkali	NaCl	
Testing conditions	Total alkali content Na_2O eq. (%)	Cement only (0.63) 1.13, 1.63, 2.13, 2.63, 3.13 (Cement (0.63) + NaCl (%))	
	Mix proportions	W/C	0.45, 0.50, 0.55
		S/C	2.25
	Dimension of specimen	4 × 4 × 16 cm	
	Items of measurement	Expansion, Dynamic Young's modulus, Compressive strength, Modulus of repture	

3. RESULTS AND DISCUSSION

3.1 Measured values

The values of the length change, strength, and dynamic Young's modulus are the mean values obtained from three specimens. The maximum difference between the maximum and minimum measured values of the length change of each of three specimens were 0.0010% for the age of one month, 0.0015% for the age of three months, 0.0018% for the age of six months, and 0.0025% for the age of one year. These values were within the limit specified in ASTM, that is, the difference between the mean value and each measured value must be less than 0.003%. The difference between the mean value and each measured value for the strength and the dynamic Young's modulus was within 10%.

3.2 Effects of the water-cement ratio

(1) Water-cement ratio
Three kinds of reactive aggregate (O, T1 and T2) were used in this test. The relationship between the water-cement ratio and the expansion of mortar speci-

mens using aggregate O at the age of 12 months is shown in Fig. 3. It can be generally said with the three aggregates that the expansion of specimens is constant or becomes lower with increases in the water-cement ratio at the range of lower total alkali content less than 1.5% (Na_2O eq.); while with a total alkali content of 2.0%, expansion is constant or becomes larger with increases in the water-cement ratio. The reason is considered to be that the diffusion coefficient of alkali ions generally becomes lower and the reaction is restrainted to a certain degree with decreases in the water-cement ratio. At the same time, the stiffness of the cement solid becomes large. This results in the pressure due to expansion tending to become small, if the reactivity is at the same level [1]. On the other hand, if the unit water content of the mortar is constant, the unit cement content becomes large with decreases in the water-cement ratio, which causes the high alkali content and increases expansion. From this point of view, it can easily be understood that there exists a pessimum value for the water-cement ratio.

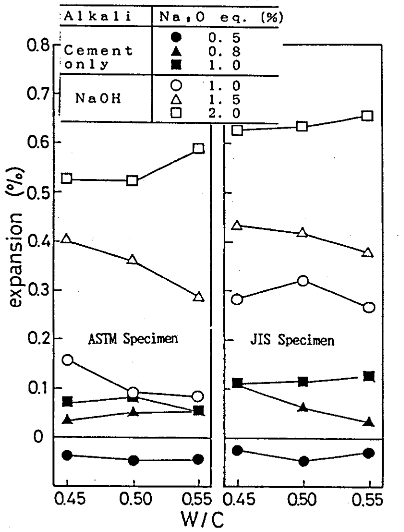

Fig. 3 Expansion versus water-cement ratio.

F.E. Jones et al [2]. pointed out that the pessimum value for the water-cement ratio was 0.40, and W.J. French [3] indicated that the expansion becomes smaller with decreases in the water-cement ratio.

We consider that the parameters which cause increases in expansion with increases in the water-cement ratio are positive parameters and that the parameters which cause decreases in expansion with increases in the water-cement ratio are negative parameters. The strength and diffusion coefficients are considered to be the positive parameters, and the ion concentration in the micro pores and the volume of the voids are considered the negative parameters. In this experiment, as described above, the effects of the water-cement ratio on the expansion were different with the change in total alkali content. Increases in the Na_2O eq. value mean increases in the ion concentration. With the Na_2O eq. value at 2.0%, the effects of the ion concentration, which is a negative parameter, become small and the effects of the decrease in the strength and diffusion coefficients, which are the positive parameters, become large. The result was that the expansion was constant or increased with increases in the water-cement ratio. On the other hand, in the lower ranges of total alkali content, Na_2O eq. less than 1.5%, the effects of decreases in the ion concentration due to increases in the water-cement ratio become large, causing a decrease in expansion. The existence of these phenomena can be recognized from the study of Diamond et al. [4], that there is a limiting value for the concentration of alkali ions in solution in the pores of mortar to cause alkali aggregate reaction and maintain its activity, and the expansion becomes small in the range of ion concentration lower than this limit.

(2) Relationship between the dimensions of the specimens and the water-cement ratio

Fig. 4 shows the relationship between the expansion of ASTM specimens and JIS specimens. In this figure, the dotted line shows JIS/ASTM=1 and the solid line shows 25, the regression line for all points (JIS/ASTM=1.25). From this figure, it can be seen that, although there is a little difference among the kinds of aggregate, the expansion of JIS specimens are greater than that of

ASTM specimens at any water-cement ratio.

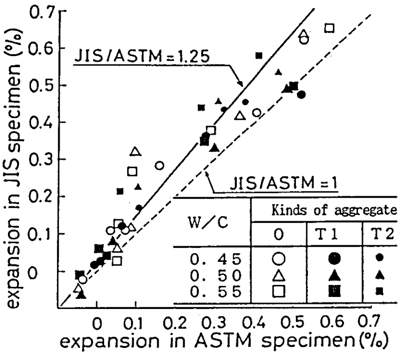

Fig.4 Effect of the size of specimen

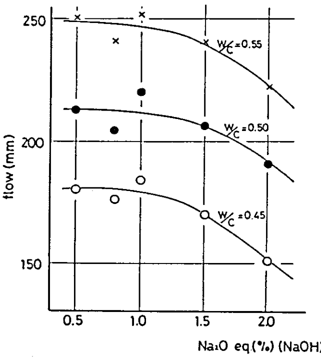

Fig.5 Relationship between flow values and total alkali content of mortar

3.3 Compacting time

It is well known that a large addition of NaOH causes hardening and reduced flowability of fresh mortar. Fig. 5 shows the relationship between the total NaOH content and the flow values of mortar indicated for each water-cement ratio. The flow value of mortar of 2.0% Na_2O eq. is 90% of that of cement only (0.5% Na_2O eq.), and it can be estimated that the amount of additional NaOH scarcely affects the compacting ability.

The expansion over time of mortar for each compacting time is shown in Fig. 6, and the relationship between the expansion and the compacting time at each age is shown in Fig. 7.

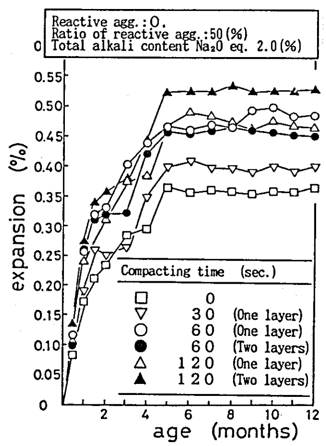

Fig.6 Effect of compacting time

From Fig. 6, the outline of the increase of expansion over time is almost the same for any compacting time, but the expansion for compacting times of 0 and 30 seconds are 0.05% less at one month and 0.05 - 0.1% less at 6 months than that for other compacting time.

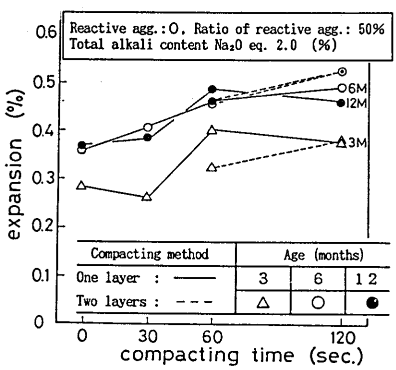

Fig.7 Expansion versus compacting time

From Fig. 7, the expansion becomes small with decreases in compacting time for the specimens cast in one layer. This is because the number of pores in mortar becomes large with decreases in compacting time and the gel produced by the reaction can penetrate into the pores, causing a decrease in the pressure of expansion. It can be considered that air entrainment with an air entraining agent is effective in reducing the expansion. This tendency was reported by Nohmachi et al [5]. and A.D. Jansen et al. [6]. For example, Jansen described that entrainment of 4% air reduced expansion by 40%. It is also reported that the use of an air entraining agent containing $CaCl_2$ may raise the reaction activity and increase the expansion [7].

The expansion of specimens cast in two layers is a little bit greater than of those cast in one layer, for long compacting times.

3.4 Expansion in alkali solution

Mortar bars containing one of the four kinds of reactive aggregates (O, T1, T2, Q) or the non-reactive aggregate were immersed into 10% solutions of NaCl, NaOH and KOH, sea water, and water at 20°C, and into water at 40°C. At the same time, mortar bars pre-cured in water at 20°C for 28 days were immersed into each solution.

The expansion over time of the mortar specimens immersed into sea water and the NaOH solution are shown in Figs. 8 and 9.

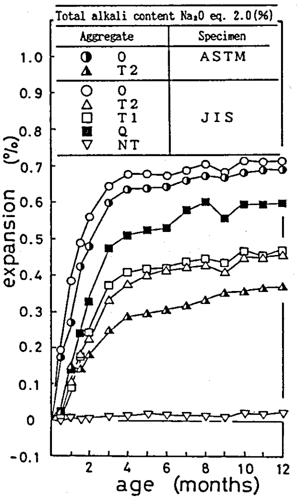

Fig. 8 Expansion versus time (Sea water)

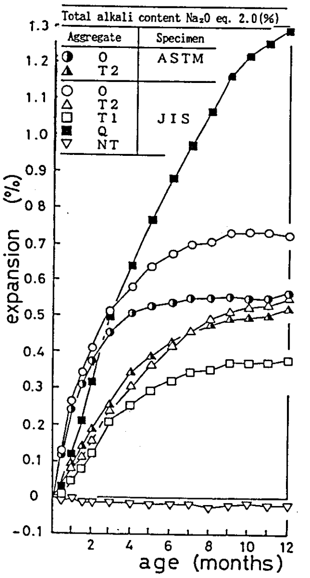

Fig. 9 Expansion versus time (10% NaOH)

In sea water, specimens made from non-reactive aggregate did not expand at all, and specimens made from O aggregate expanded 0.3 - 0.4%, at the age of one month, larger than that of specimens containing any of the other reactive aggregates. The expansion of almost any specimen containing reactive aggregate reached a constant by the age of 4 - 5 months. The increase in expansion after that was only 0.05 - 0.10%.

In the 10% NaOH solution, the mortar specimens made from Non-reactive aggregate did not expand at all. On the other hand, the all of the specimens made from reactive aggregates expanded. The expansion of the specimens tended to be constant by the age of 7 months, except for those using the reactive aggregate Q. The expansion of specimens made from reactive aggregate Q increased linearly over time until the age of 9 months. After that, the rate of increase tended to reduce slightly. The expansion of specimens immersed in the NaCl solution was 1.5 - 1.8 times larger than that immersed in sea water. The expansion of specimens in the KOH solution was almost the same as that in the NaOH solution.

Thus, the specimens made from non-reactive aggregate did not expand in any solution, while the expansion of specimens made from any of the reactive aggregates reached a constant value by the age of 4 months. The specimens made from reactive aggregate O expanded rapidly, and reached constant expansion value in the early age, which with reactive aggregate Q, expansion increased linearly in the 10% solutions of NaCl, NaOH and KOH. The expansion of specimens made from reactive aggregates T1 or T2 was smaller than that made from other reactive aggregate. As described above, the expansion characteristics of specimens was different depending on the kind of aggregate used in the mortar.

The expansion over time of specimens made from reactive aggregates O and T1 are shown in Fig. 10 and 11. From these figures, it is clear that the expansion of specimens immersed in the 10% NaCl solution was 1.8 - 2.5 times larger than that of specimens immersed in the other solutions, at the age of 12 months. Except immersion in 20°C water, the expansion continues even after 7 months in the 10% NaOH or KOH solution, while the expansion of specimens immersed in sea water or 10% NaCl solution tends to be rapid in the early age and to reach a the constant value at the relatively early age. The specimens immersed in sea water or alkali solution showed larger expansion compared than those tested by the conventional mortar bar method. Comparing the results obtained from the test of immersed in 40°C water with the conventional mortar bar method, expansion grows rapidly and reachs a constant by 1 - 4 months of age in the former test, while in the later test, expansion grows rather slowly and exceeds the value of former test by the age of 3 - 4 months, becoming about 0.15% lager by the age of 12 months.

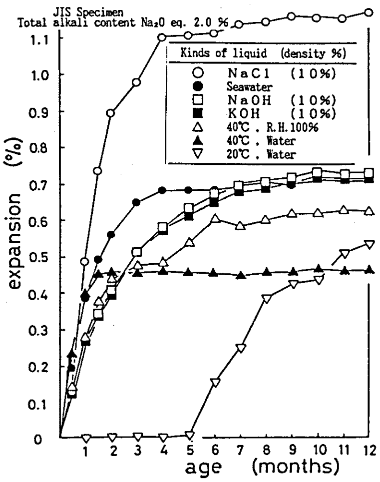

Fig.10 Expansion versus time
(Aggregate : O)

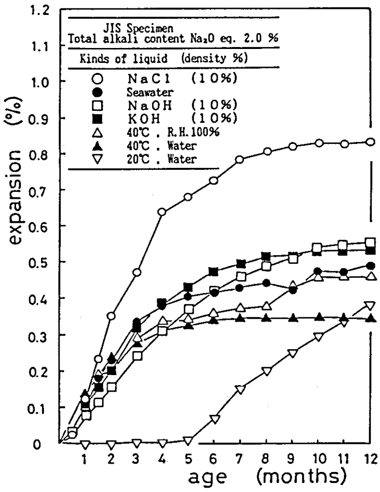

Fig.11 Expansion versus time
(Aggregate : T 2)

The specimens immersed in 20°C water do not expand until the age of 5 months. After that, the expansion characteristics are similar to those of the conditions of 40°C, R.H.100 %. At the age of 12 months, the expansion of the specimens immersed in 20°C water reaches 80% of the condition of 40°C, R.H.100% specimens and continues expanding. It is considered that both values of expansion become the same.

It is clear that expansion develops most in the NaCl solution. Even in sea water, which is only 3.6% salt concentration, expansion develops to some extent. This shows that the solution containing Cl^- magnifies the expansion due to alkali aggregate reaction. The fact that the expansion continues beyond the age of 7 months in NaOH and KOH solutions shows that expansion develops over a long a long period in solution containing OH^-. From the results obtained from the 40°C water immersion test and the conventional mortar bar test(40°C, R.H.100%), it was seen that,in the 40°C water immersion, a large amount of expansion develops in the early age because of a sufficient water supply, but as the alkali component in mortar and/or gel produced by reaction runs off to the water, the maximum expansion value becomes smaller than that of the conventional mortar bar test. With immersion in water at 20°C, the time when the expansion begins is considerably later, but the final values of expansion seems to become almost the same as those of immersion in water of 40°C or 40℃, R.H.100%.

The mortar specimens made from the cement with an alkali content of 0.5% as Na_2O eq. were immersed into several kinds of liquids just after demolding (0.5% specimens) and after pre-curing for 28 days in water at 20°C (0.5% pre-curing specimens). Fig. 12 shows a sample of expansion characteristics obtained from this test.

0.5% specimens did not expand in sea water at all, but in the alkali solution, expansion began occur by the age of one month, and increased rapidly. The period in which the expansion was growing was only 3 - 4 months. The specimens which were pre-cured in water at 20°C showed almost the same expansion characteristics as those with no pre-curing. The expansion of these specimens were only half of those with no pre-curing. With the NaCl solution, the final values of expansion became large with increases in the alkali content of Na_2O eq., while in the NaOH and KOH solutions, the final values of expansion were almost the same regardless of the alkali content.

The specimens having an alkali content of 2.0% as Na_2O eq. started to expand the earliest, followed by the 0.5% specimens, and then by the 0.5% pre-cured specimens. In the solutions of NaCl, NaOH, and KOH, even the 0.5% pre-curing specimens expanded by more than 0.1% by the age of 7 months, which is considered deleterious expansion. On the other hand, in sea water, neither the 0.5% specimens nor the 0.5% pre-curing specimens showed deleterious expansion.

The above can be summarized as follows:

1) The expansion characteristics of mortar in alkali solution differ with the kind of aggregate.

2) Solutions for immersion containing Cl^- cause larger expansion than solutions containing OH^-.

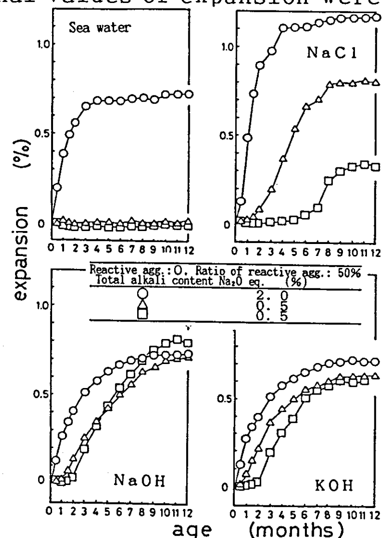

Fig. 12 Expansion versus time
(Effects of pre-curing in water)

3) Expansion occurs for a long time in solutions containing OH^-.

4) Expansion starts, and reaches a constant value, earlier in water at 40°C than in the conventional mortar bar test (40°C, R.H.100%). The final values of expansion are less in water at 40°C than those of the conventional mortar bar test.

5) In water at 20°C, the time when expansion starts is rather late, but the final values of expansion seem to become almost the same as those in water at 40°C or in the conventional mortar bar test.

6) The specimens containing only 0.5% alkali as Na_2O eq. expand deleteriously in the 10% solution of alkali compound.

7) The specimens mentioned above do not expand deleteriously in sea water.

8) Pre-curing in water at 20°C for 28 days is effective in delaying the time when expansion starts, but does not reduce the final values of expansion.

From the results described above, a rapid method can be proposed for determining the reactivity of aggregate by immersion into a solution of alkali and water.

Chatterji in Denmark [8] proposed a rapid method wherein a specimen of mortar having a mix proportion of C:S=1:3 is immersed into a saturated NaCl solution at 50°C.

From the results of this experiment, it became clear that the mortar specimen expanded maximally when immersed into a NaCl solution, and this expansion was increased with the addition of NaOH to the mortar. Therefore, as a rapid method of immersion into alkali solution, the following conditions seem to be proper: The total alkali content of mortar should be adjusted to 1.5 - 2.0% as

Na_2O eq. by the addition of NaOH, and the specimens of mortar should be immersed into a NaCl solution to accelerate the rate of expansion. By this test method, abnormal expansion of 0.1 - 0.3% appears at the age of one month, and it is possible to determine the reactivity of a aggregate within a short period.

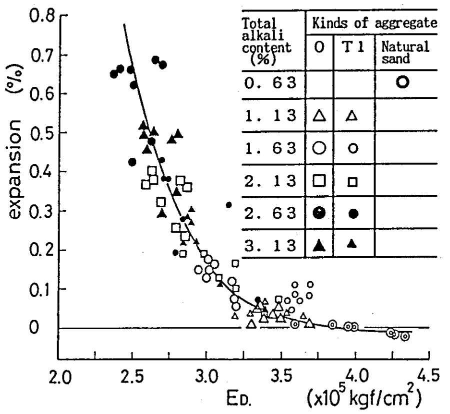

Fig. 13 Relationship between expansion and dynamic Yong's modulus

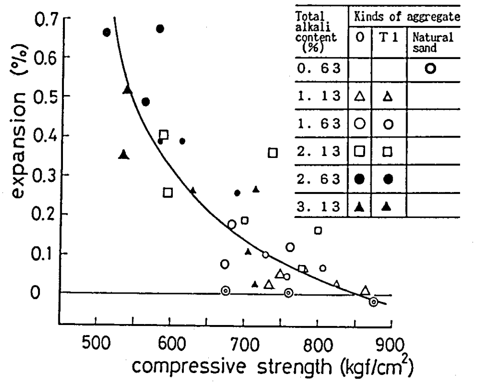

Fig. 14 Relationship between expansion and compressive strength

3.5 Relationship between expansion and strength and dynamic Young's modulus

The dynamic Young's modulus and the modulus of rupture and compressive strength were measured using JIS specimens made from reactive aggregates O and T1.

The relationships between the expansion of mortar and these measured values are shown in Fig. 13 - Fig. 15. From Fig. 13, the dynamic Young's modulus of each mortar is seen to decrease rapidly with increases of expansion, while from Fig. 14 and 15, the compressive strength and the modulus of rupture of the mortar also decrease with increases in expansion.

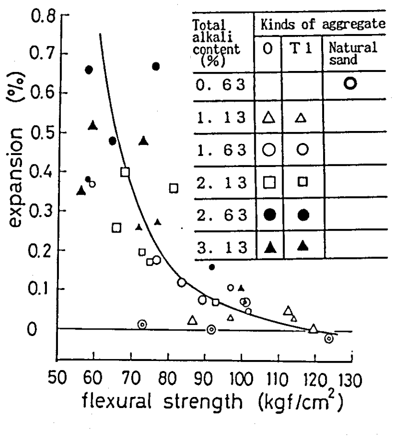

Fig. 15 Relationship between expansion and modulus of rupture

It has been reported that concrete made from reactive aggregate has a compressive strength and dynamic Young's modulus of 83% of that made from non-reactive aggregate. For tensile strength, this ratio is 67% [9].

This shows that the rate of expansion due to alkali aggregate can be estimated by the rate of decrease of the mechanical properties of mortar.

As described above, there is a negative correlation between the expansion of mortar and the modulus of rupture, the compressive strength and the dynamic Young's modulus. Thus, it became clear that the modulus of rupture, the compressive strength, and the dynamic Young's modulus of mortar decrease with increases in the expansion of the mortar.

4. CONCLUDING REMARKS

In this study, an investigation into various factors which should be considered for applying the ASTM mortar bar test method to aggregate produced in Japan was carried out. The factors adopted in this study were the water-cement ratio, the compacting method, immersion into alkali solution, and the effects of these factors on the expansion of mortar. At the same time, the relationship between expansion and changes in mechanical properties due to alkali aggregate reaction were examined.

The conclusions of this study may be summarized as follows:

(1) Water-cement ratio and the dimensions of the specimens.

1) With the cement content fixed to a constant, the expansion characteristics of mortar were affected by the total alkali content. The expansion of mortar is constant or decreases with increases in the water-cement ratio in the area of low alkali content less than 1.5% as Na_2O eq., but is constant or increases with increases in the water-cement ratio in the area of alkali content of 2.0% as Na_2O eq..

2) The expansion of the JIS specimens was larger than that of the ASTM specimens, regardless of the water-cement ratio and the aggregate-cement ratio.

(2) When the compacting time was long, i.e., the compacting was sufficient, the expansion of mortar became large.

(3) When specimens were immersed into a solution of alkali compound, the expansion was small in the solution containing OH^- than those in the solution containing Cl^-, but the expansion continued for a longer time. Specimens where the total alkali content, as Na_2O eq. was 0.5% showed a deleterious expansion of greater than 0.1% in a 10% alkali immersion. The specimens in sea water or water at of 20°C showed the possibility expansion in a total alkali content of more than 1.0% as Na_2O eq..

(4) There is a negative correlation between the expansion of mortar and the modulus of rupture, the compressive strength and the dynamic Young's modulus. Thus, the mechanical properties, such as the strength and the dynamic Young' modulus, become lower with increases in expansion.

From these results, the following conditions can be proposed for the mortar bar method to determine the potential reactivity of aggregate in Japan. Mixing, compacting, and dimensions of the mortar specimen are to be conducted according to JIS R5201, and the water-cement ratio is to be set to 0.45 - 0.50, the sand-cement ratio to 2.25, (equal to ASTM standard), and the total alkali content to 1.5 - 2.0 as Na_2O eq., using NaOH.

In the future, the authors plan to carry out a study of the relationship between the expansion of mortar and concrete, in order to develop a test method for the evaluation of alkali aggregate reaction in concrete specimens.

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