Effect of Gypsum Content in Cement on the Autogenous Shrinkage of Low-Heat Portland Blast-Furnace Slag Cement Concrete

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by

Katsuki Harada* and Hiromichi Matsushita**

Abstract

When a low-heat portland blast-furnace slag cement is used, the thermal expansion strain of the concrete shows reversal shrinkage in spite of a continuous temperature rising as a result of abrupt autogenous shrinkage far greater than the thermal expansion. The autogenous shrinkage strain was studied in terms of gypsum content in cement, and was able to be reduced by increasing gypsum content in cement. Prevention of the early-age rapid hydration development of granulated blast-furnace slag at high temperature was also confirmed. Moreover, the amount of transition from ettringite to monosulfate of a hydration product was determined, and the result was related with autogenous shrinkage. Because aluminate hydrates are most affected by gypsum.

Keywords : Thermal expansion strain, Autogenous shrinkage strain, Gypsum content, Degree of slag hydration, Transition from ettringite to monosulfate

1. Introduction

Various types of low-heat cements have been used to reduce the thermal cracking of mass concretes. These include a cement with a reduced heat of hydration release at early ages and a blended cement substituted with a quality admixture such as granulated blast-furnace slag and fly-ash[1],[2]. However, it has been reported that the blast-furnace slag may promote the autogenous shrinkage[3],[4], and the autogenous shrinkage has been a problem with the use of recent high-strength concretes[5]. Another report recommended to pay attentions to autogenous shrinkage when portland blast-furnace slag cement is used in the mass concrete constructions[6], while the autogenous shrinkage behavior of concrete after a temperature history due to cement hydration is still unknown. In our previous paper[7], early-age strain of concretes made of various types of portland blast-furnace slag cement were determined taking into account the heat of hydration. As a result, it was shown that the

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thermal expansion strain of concretes, using moderate-heat and low-heat portland blast-furnace slag cements, showed reversal shrinkage even in a continuous temperature rising. This accelerated autogenous shrinkage was attributed to reduction of pore diameter associated with the transition of a hydration product, from ettringite to monosulfate. Effect of gypsum content in cement was found to be significant in such a manner that the autogenous shrinkage strain was reduced with an increase in gypsum content. This was confirmed by experiments with hardened cement paste specimens at a temperature of 20°C. However, the autogenous shrinkage depends strongly on temperature and shows high reactivity at elevated temperatures. Thus factors affecting the autogenous shrinkage at a room temperature should be further examined in terms of temperature history. Because the reduction of pores diameter is associated with the transition from ettringite to monosulfate, amount of ettringite transition should also be determined.

In this paper, the low-heat portland blast-furnace slag cement that exhibited significant autogenous shrinkage according to temperature changes was dealt with. Effect of gypsum (SO_3) content in cement on the autogenous shrinkage was examined by determining the autogenous shrinkage strain under hydration temperature histories. Because aluminate hydrates are most affected by gypsum. Next, behavior of aluminate hydrates in various types portland blast-furnace slag cement at a high temperature (40°C) was detected by X-ray diffraction and the amount of transition from ettringite to monosulfate was determined. And the result was related with the reduction of pore diameter and autogenous shrinkage strain.

Table 1 Used materials

Materials	Properties	Mark
Portland blast-furnace slag cement type B	Density:3.04g/cm^3,Blaine value:4120cm^2/g Blaine value of slug:4000cm^2/g,Admixture content of slug:40%	BB
Moderate-heat portland blast-furnace slag cement	Density:3.04g/cm^3,Blaine value:3800cm^2/g Blaine value of slug:4000cm^2/g,Admixture content of slug:55%	MB
Low-heat portland blast-furnace slag cement	Density:3.03g/cm^3,Blaine value:4720cm^2/g Blaine value of slug:6000cm^2/g,Admixture content of slug:60%	LB
Low-heat portland cement	Density:3.04g/cm^3,Blaine value:4120cm^2/g	L
sea sand	Density in saturated surface-dry condition:2.55g/cm^3,F.M:2.13	S
crushed sand	Density in saturated surface-dry condition:2.66g/cm^3,F.M:2.69	
crushed stone 1305	Density in saturated surface-dry condition:2.73g/cm^3,F.M:6.16	G
crushed stone 1305	Density in saturated surface-dry condition:2.73g/cm^3,F.M:7.02	
Air-entraining and water-reducing admixture	Lignin sulpho acid	Ad
Superplasticizer	Polycarboxylic acid	Sp

2. Early-age strain properties of portland blast-furnace slag cement concrete

As reported in the previous paper[7], thermal expansion strain under temperature histories due to heat of hydration and autogenous shrinkage strain of portland blast-furnace slag cement type B (BB), moderate-heat portland blast-furnace slag cement (MB) and low-heat portland blast-furnace slag cement (LB) are shown in **Fig. 1** and **Fig. 2**. Used materials, mix proportions of concrete and chemical composition of cements are shown in **Table 1**, **Table 2** and **Table 3**. It is shown in **Fig. 1** that the thermal expansion strain showed reversal shrinkage in spite of a continuous temperature rising, and this tendency was most remarkable when LB is used. When BB was used, however, the reversal shrinkage was not observed though an increase in thermal expansion strain was temporarily delayed at a change of

Table 2 Mix proportions of concrete

W/C (%)	s/a (%)	Unit content(kg/m^3)				Ad	Slump (cm)	Air content (%)
		C	W	S	G			
46.1	41.9	330	152	757	1100	C×0.25%	8±2.5	4.5±1.0

Table 3 Chemical composition of cements

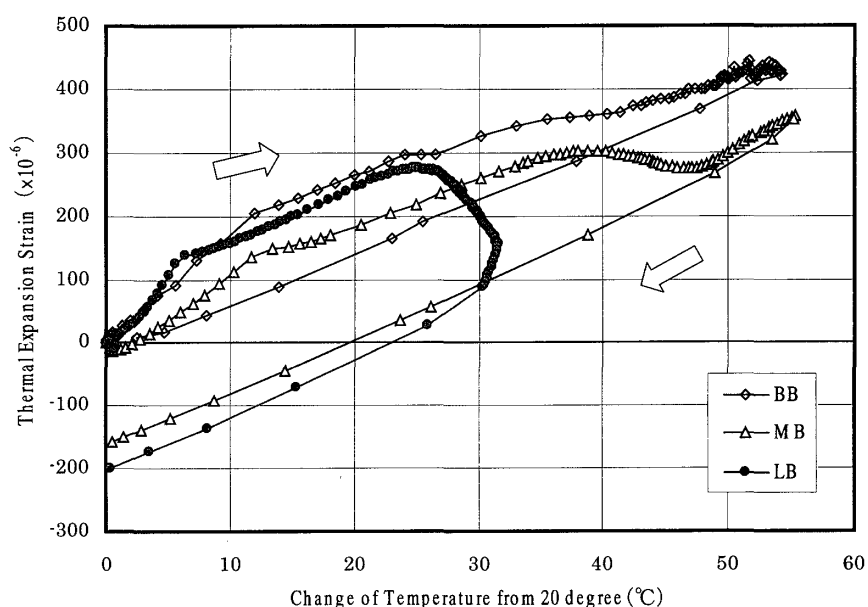| Cement	Chemical composition(%)								Mineral composition(%)			
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	C_3S	C_2S	C_3A	C_4AF
BB	25.32	9.07	1.84	55.02	3.93	2.15	0.30	0.31	-	-	-	-
MB	28.17	10.60	1.78	50.71	4.70	2.30	0.28	0.31	-	-	-	-
LB	29.35	10.66	1.68	49.44	4.60	1.90	0.26	0.30	-	-	-	-
N	21.34	5.40	2.60	65.14	1.59	2.04	0.35	0.36	57	18	10	8
M	24.31	3.51	3.36	63.20	1.24	2.44	0.32	0.39	37	42	4	9
L	25.94	3.25	3.23	62.46	1.03	2.02	0.26	0.31	25	56	3	10

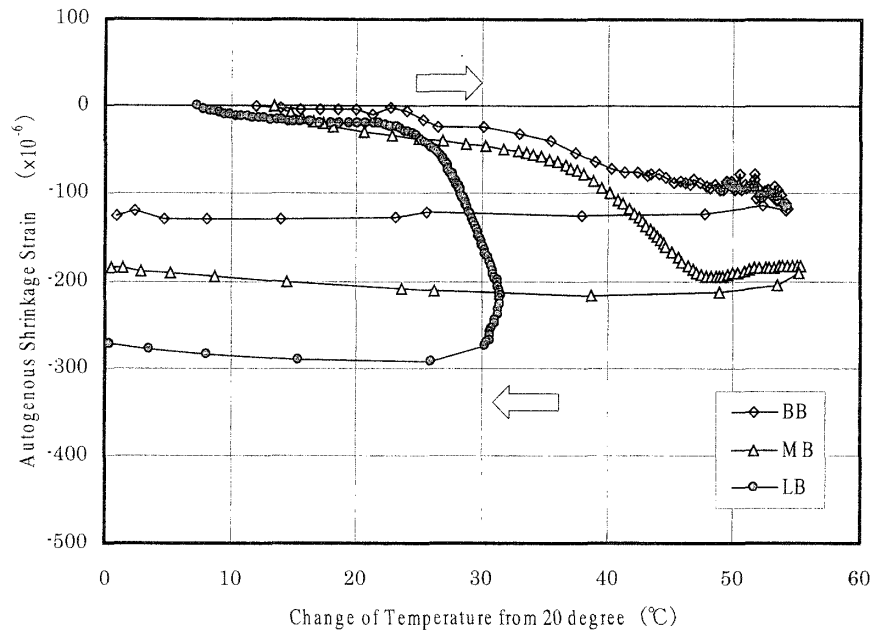**Fig. 1** Thermal expansion strain of various type of portland blast-furnace slag cement

Fig. 2 Autogenous Shrinkage strain of various type of portland blast-furnace slag cement
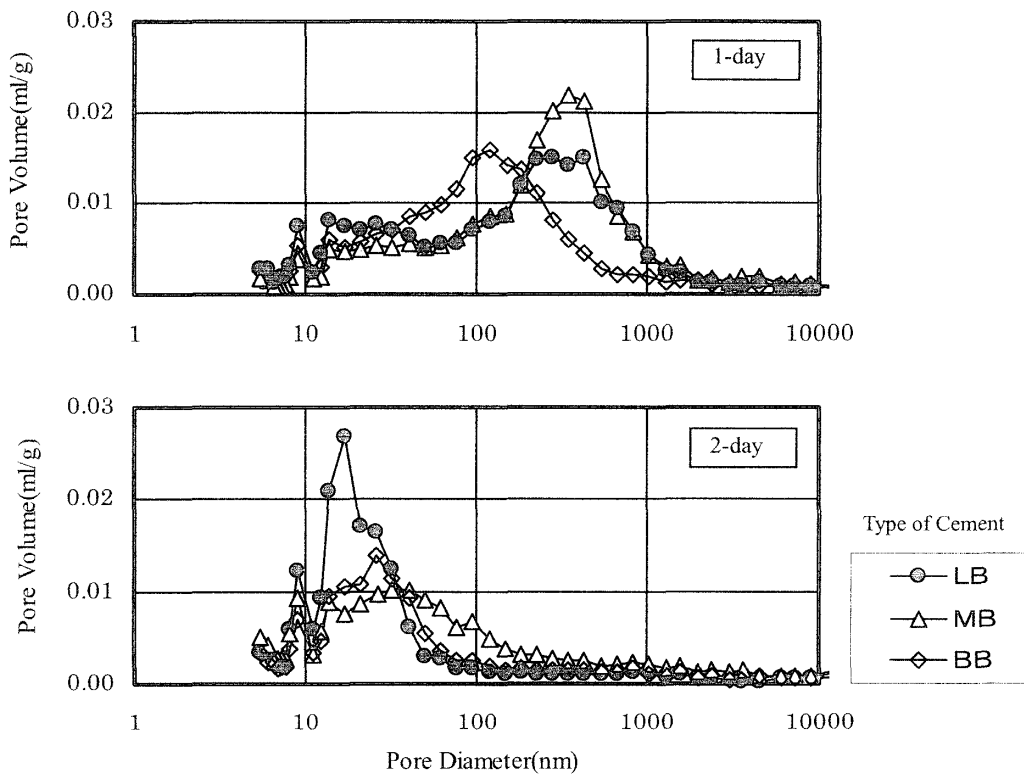

Fig. 3 Pore size distribution of various type of portland blast-furnace slag cement paste

temperature range from 30 to 40°C.

When MB and LB were used, a critical point was observed where an abrupt increase in autogenous shrinkage and decrease in thermal expansion strain were present at the same time as shown in **Fig. 2**, and this could be a cause of the reversal thermal expansion strain. When BB was used, no abrupt autogenous shrinkage strain was observed. At the same age as the abrupt increase of autogenous shrinkage strains were observed, 3.82-day for MB and 1.79-day for LB, pore diameter distributions showed reduction with a marked peak at 50 nm
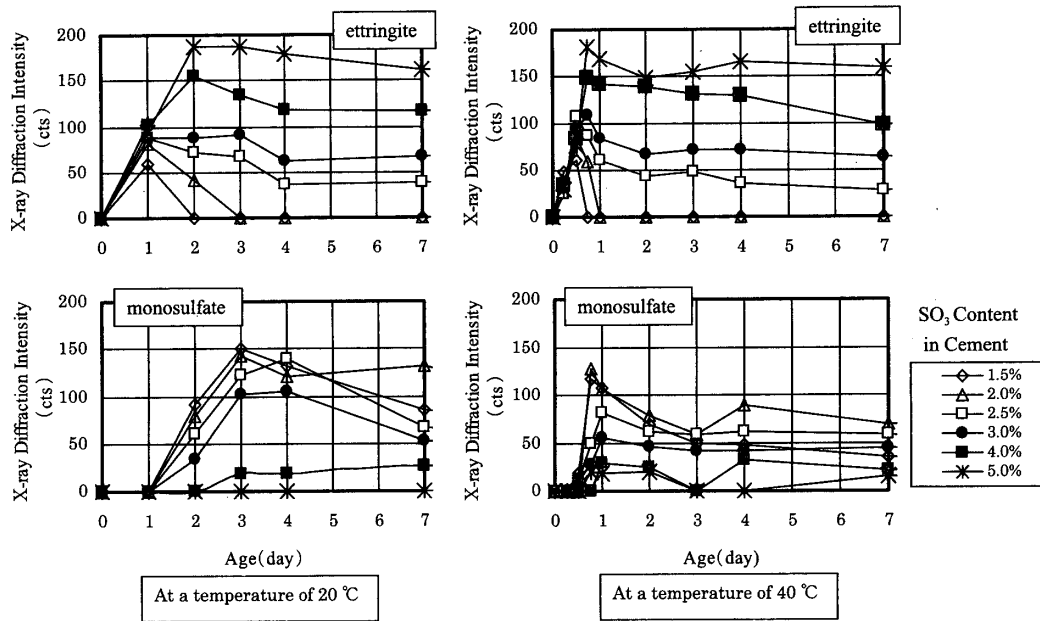
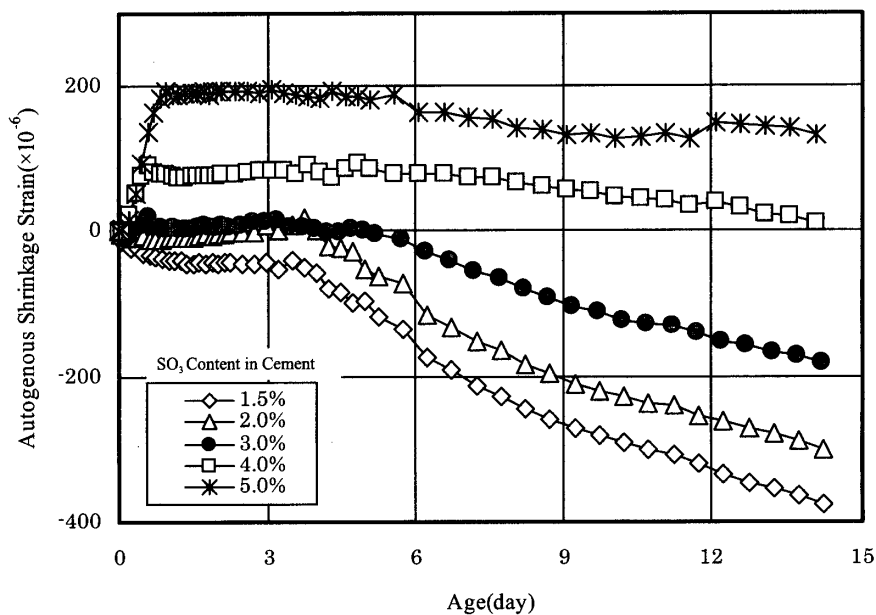**Fig. 4** Results of the X-ray diffraction

Fig. 5 Effect of SO₃ content in LB cement on the autogenous shrinkage of paste at a temperature of 20°C

or less. This abrupt pore reduction can be a cause of the abrupt autogenous shrinkage strain. As an example of the abrupt pore reduction, pore size distribution of LB cement paste is shown in **Fig. 3**. An X-ray diffraction analysis of aluminate hydrates at a very early stage of hydration was executed and a working mechanism was proposed that the ettringite-monosulfate transition affects the pore reduction and the transition can be reduced by increasing gypsum content in cement (hereafter referred to as SO₃ content). Results of the X-ray diffraction is shown in **Fig. 4**, where the data of SO₃ content of 2.0 percent corresponds to the cement used in concrete experiments. Effect of SO₃ content in LB cement on the autogenous shrinkage strain of cement paste was determined and the reduction of the autogenous shrinkage strain with an increase in SO₃ content is shown in **Fig. 5**.

3. Effects of gypsum in cement on the autogenous shrinkage strain

3.1 Outline of experiments

(1) Behavior of aluminate hydrates in the various portland blast-furnace slag cement pastes

Each of the BB, MB and LB cement pastes with a water-cement ratio of 1.0 was prepared in a 30-mililiter plastic beaker with a cap and cured in a 40-degree drying oven until the ages for testing. No admixture was used to solely compare the hydration characteristics of the cements. When the specimens reached a specified age for testing, they were removed from the beaker, rapidly crushed in a mortar into a 2.5 to 5.0 mm in diameter and interrupted hydration in an acetone container. Subsequently, specimens were ground together with acetone in a disk-mill, ltered under a reduced pressure and freeze-dried the portion that passed a 45-micrometer sieve. The freeze-drying was a vacuum drying of specimen frozen within 2-hour. Ettringite and monosulfate in a specimen with age of 1, 2, 3, 4 and 7-days were detected by X-ray diffraction, where the amount of ettringite were determined at the age when diffraction intensity showed a peak. The determination of ettringite was made by a stepwise addition of a pure ettringite, synthesized from hydrogarnet and dihydrated gypsum, to the specimen which is then subjected to X-ray diffraction. The resulting diffraction intensities were plotted against ettringite contents and an X-axis intersection point of the linear regression of the plot gave the original ettringite content. Outline of the determination of ettringite is shown in **Fig. 6.**

(2) Effects of gypsum (SO_3) content in cement on the thermal expansion strain of concretes

Used materials and mix proportions of concrete in this experiment are nearly identical with those described in the previous section and are shown in **Table 1, Table 2** and **Table 3**, but only LB cement was used with a SO_3 content of 2.0, 2.5 and 3.0 percent. Specimens for the determination of thermal expansion strain are shown in **Fig. 7.** An embedded electric strain gauge transducer was KM-100B type with a temperature sensor and elastic modulus of 40 N/mm^2, and was placed in the center of a steel cylindrical mold with a diameter of 40 cm and a height of 40 cm. Concrete was placed to completely fill the mold and was capped by a steel plate. The temperature of the mixed up concrete was 20°C. The specimens were placed in an adiabatic temperature rise apparatus, where temperature changes of specimens
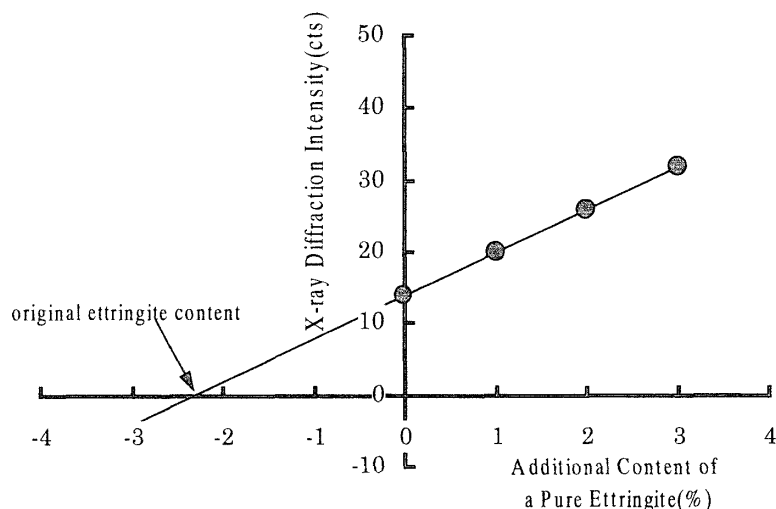

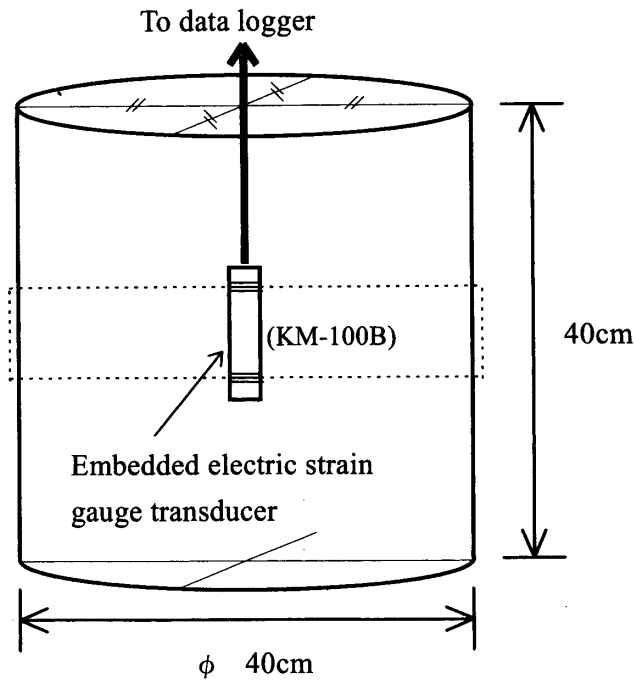**Fig. 6** Outline of the determination of ettringite

To data logger

(KM-100B)

40cm

Embedded electric strain
gauge transducer

φ 40cm

Fig. 7 Specimens for the determination of thermal expansion strain

SO₃ Content in Cement

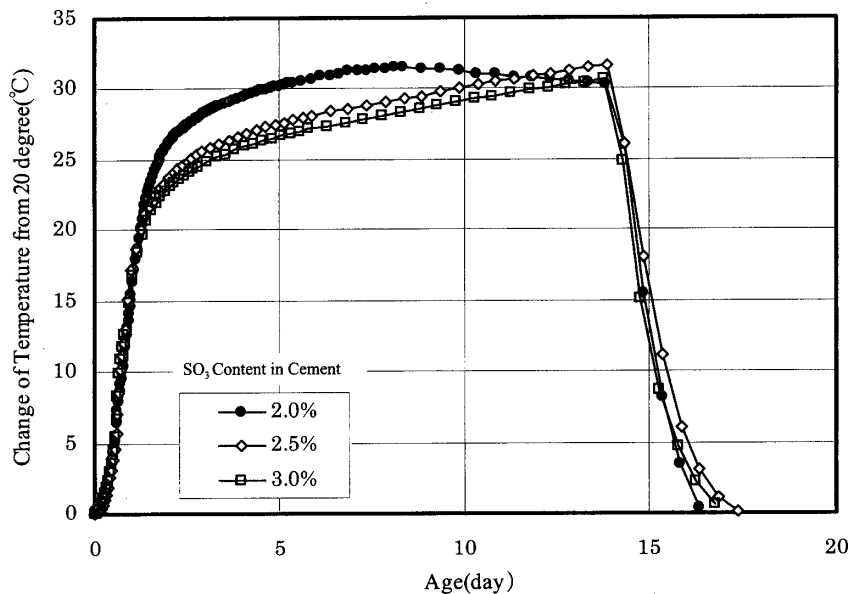—●— 2.0%
—◇— 2.5%
—☐— 3.0%

Age(day)

Fig. 8 Temperature histories of specimens with ages

due to heat of hydration can be followed by the atmospheric temperature, and subjected to each temperature history without external effects. The temperature rise of the specimens had almost come to the end at 14-days and the test was terminated. Temperature histories of specimens with ages are shown in **Fig. 8**.

(3) Effects of gypsum (SO_3) content in cement on the autogenous shrinkage strain of concretes

Used materials and mix proportions of concrete in this experiment are the same as the thermal expansion strain experiment. Specimens for the determination of autogenous shrinkage strain are shown in **Fig. 9**. A teflon sheet and a polystyrene board were placed in a steel mold of $10 \times 10 \times 40$ cm in size to allow young concrete for free volume changes without restriction of the mold. An embedded electric strain gauge transducer KM-100B is
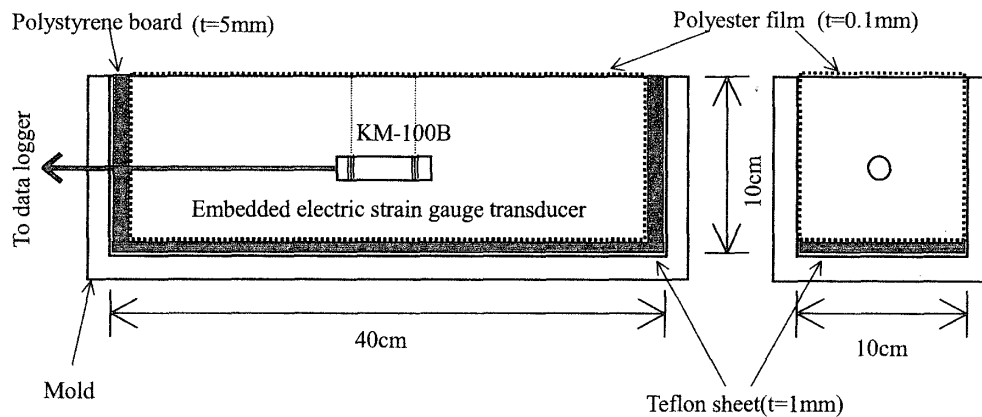
Fig. 9 Specimens for the determination of autogenous shrinkage strain

placed in the center of the mold in the same way as the case of thermal expansion strain measurement. Concrete with a mixed up temperature of 20°C was placed in the mold and sealed with a polyester film. Specimens were then set, together with the thermal expansion strain specimens, in the adiabatic temperature rise apparatus and subjected to each temperature history as shown in **Fig. 8**. They were demolded at the age of 2-days and thereafter sealed with an aluminum sticky tape and returned to the apparatus to continue the test.

The reference length of the autogenous shrinkage measurement was defined as the length when the skeleton of a hardened cement paste was formed, which can be observed as the first moment when a temperature rise increment become lower than that of immediately before. Because a measured thermal expansion strain comprises an autogenous shrinkage strain and a pure thermal expansion strain[8], the autogenous shrinkage strain can be obtained by subtracting pure thermal expansion strain, calculated using the center temperature of the $10 \times 10 \times 40$ cm specimen, from the measured thermal expansion strain. The coefficient of thermal expansion was obtained form the relationship between the temperature decrease and corresponding strain after the termination of the apparatus at the age of 14-days. In this condition, the autogenous shrinkage strain may be excluded from the measured strain because the temperature rise, and associated hydration, almost came to the end and the test was made in a short duration (3-days). The coefficient of thermal expansion may vary according to the age but it becomes nearly constant after the final set, the moment of hardened cement paste's skeleton formation[9],[10]. Thus the coefficient of thermal expansion determined in this study is appropriate from a practical standpoint and was 9.6×10^{-6}, 8.4×10^{-6} and 8.6×10^{-6} per degree for a concrete with an SO_3 content in cement of 2.0, 2.5 and 3.0 percent respectively.

(4) Effects of gypsum (SO_3) content in cement on the degree of hydration of granulated blast-furnace slag (BS)

It is generally agreed that granulated blast-furnace slag (hereafter referred to as BS) in a low-heat portland blast-furnace slag cement greatly affects the autogenous shrinkage strain of concrete. Thus effects of SO_3 content in cement on the hydration of BS should also be studied when the autogenous shrinkage strain is studied under variable SO_3 content in cement.

The specimen had a dimension of 5 cm in diameter and 10 cm in length and was made using LB cement with a water-cement ratio of 0.461 and an admixture dosage of 0.25 percent of cement weight, which were all the same as those of concrete experiments. Sealed curing was applied in two chambers at a temperature of 20 and 40°C and then treated at each age
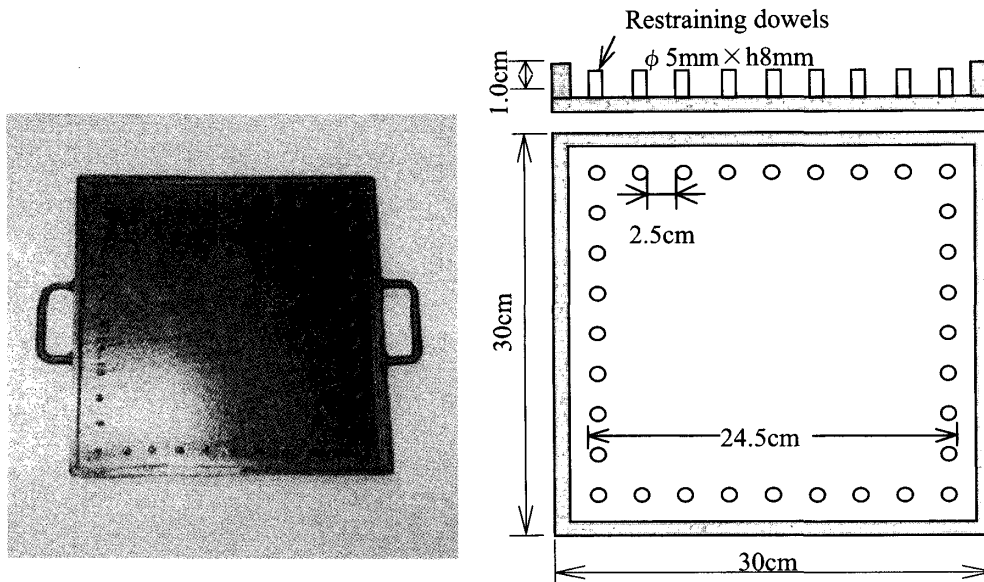
Fig.10 Outline of the mold for the observation of cracks

in the same way as done in the X-ray diffraction analysis. Test method was executed according to EDTA selective dissolution method of the concrete standard specification JSCE-D 501-1999. Hydrated cement and BS as well as unhydrated cement were dissolved into EDTA-triethanol solution leaving the unhydrated slag. Amount of hydrated slag was calculated by subtracting the unhydrated slag from the total amount of slag that applied to the cement. The degree of slag hydration was defined as the proportion of the amount of hydrated slag to the total amount of slag. Test was continued until the age of 7-days.

(5) Observation of cracks according to a change of gypsum (SO₃) content in cement
     LB cement paste with variable SO_3 content was placed in a steel flat mold, as shown in **Fig.10**, sealed with a transparent polyester film and set under a constant temperature of 20°C. The water-cement ratio was 0.3 for fear of bleeding that might have occurred with the water-cement ratio of 0.461 used in the concrete experiments. A polycarbonic acid type superplasticizer was used to have a sufficient fluidity to fill the space between restraining dowels. Visual inspection of cracks was executed once a day.

3.2 Result and discussion

(1) Behavior of aluminate hydrates in the various portland blast-furnace slag cement pastes
     X-ray diffraction of ettringite and monosulfate of BB, MB and LB cement pastes at each age are shown in **Fig.11**. Maximum diffraction intensity of ettringite was found at the age of 1-day in cases of BB and MB and 2-days for LB, while those of MB and LB disappeared at the age of 3-days. Monosulfate showed a diffraction intensity complement to that of ettringite implying the transition from ettringite to monosulfate. Because the molar volume of ettringite is about twice as large as that of monosulfate[11], pore space should increase when the transition occur in the beginning but will soon shrink due to compressive stress such as capillary tension leading to an increase in autogenous shrinkage strain. Although the ettringite-monosulfate transition was confirmed in BB cement paste, reduction of pore diameter was not observed and autogenous shrinkage didn't show such a rapid increase as seen in MB and LB. It is most likely that, in BB cement paste, the degree of transition and associated volume change were so small leading to the slight shrinkage. Because the result of X-ray diffraction can only detect the amount of well-crystallized portion, and not the entire
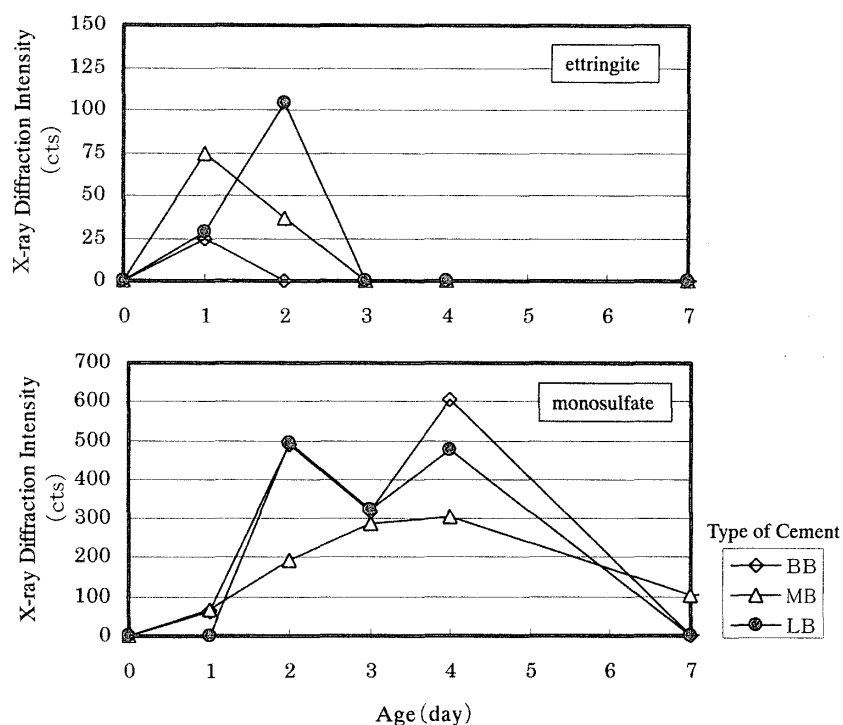
Fig.11   Results of the X-ray diffraction at a temperature of 40°C

amount of the target component, a direct determination of the amount of ettringite transition is necessary to assess the compressive stress effect to pore. Amount of ettringite at the maximum X-ray diffraction intensity, i.e. the maximum amount of transition, was determined for BB, MB and LB pastes as shown in **Fig.12**. Amount of ettringite transition by the maximum diffraction intensity of each cement was 4.61, 7.85 and 10.9 percent for BB, MB and LB respectively. It can be concluded that the relatively small pore reduction and resulting autogenous shrinkage strain of BB can be attributed to the amount of ettringite transition which is almost a half or less than those of the other portland blast-furnace slag cements.

(2)   Effects of gypsum (SO_3) content in cement on the thermal expansion and autogenous shrinkage strain of concretes

Temperature histories of concrete due to heat of LB cement hydration having variable SO_3 content are shown in **Fig. 8** in the previous section of this paper. No particular difference was observed in the temperature histories of all the ages of cements with a SO_3 content of 2.5 and 3.0 percent. However at a SO_3 content of 2.0 percent, temperature increased quicker than those of the other started at the age of 1.5-days and lasted until 14-days resulting in a rapid convergence. This shows that inuence of the transition from ettringite to monosulfate, as shown in **Fig. 5** of section 2, was still relevant in the cement with a SO_3 content if 2.0 percent and heat of hydration during monosulfate formation affected the temperature history.

Thermal expansion and autogenous shrinkage strain under temperature histories, as shown in **Fig. 8**, are plotted against temperature changes and shown in **Fig.13** and **Fig.14**. The reversal thermal expansion strain during a temperature rise was observed in specimens of any SO_3 content. However, the gradient of the curves became gentle after the reversal point for specimens with a SO_3 content more than 2.5 percent, and the shrinkage strain when the concrete temperature returned to 20°C was almost a half of that with 2.0-percent SO_3 content. The autogenous shrinkage strain of concrete with a SO_3 content more than 2.5
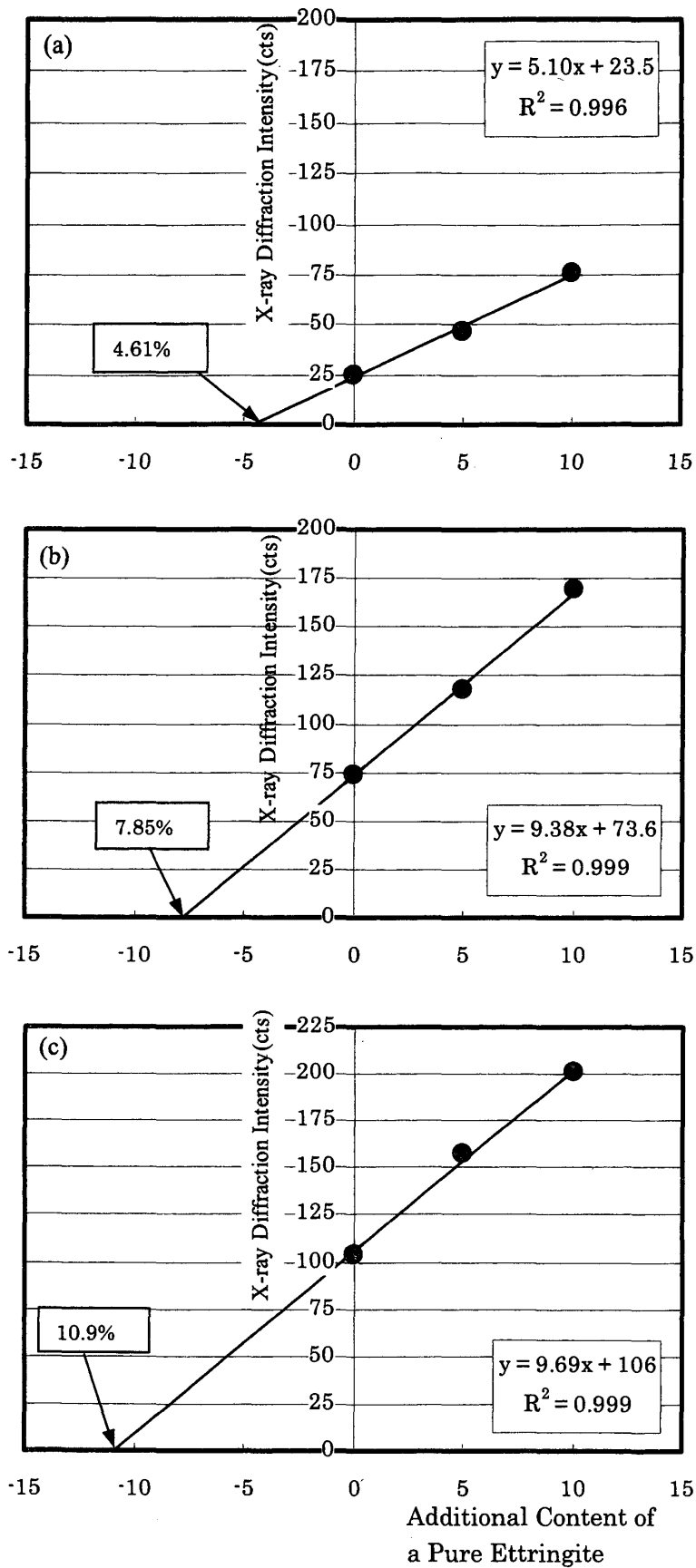
(a)

$y = 5.10x + 23.5$
$R^2 = 0.996$

4.61%

X-ray Diffraction Intensity(cts)

-15   -10   -5   0   5   10   15

(b)

$y = 9.38x + 73.6$
$R^2 = 0.999$

7.85%

X-ray Diffraction Intensity(cts)

-15   -10   -5   0   5   10   15

(c)

$y = 9.69x + 106$
$R^2 = 0.999$

10.9%

X-ray Diffraction Intensity(cts)

-15   -10   -5   0   5   10   15

Additional Content of
a Pure Ettringite

Fig.12 Results of the determination of ettringite
(a) ; In case of portland blast-furnace slag cement Type B
(b) ; In case of moderate-heat portland blast-furnace slag cement
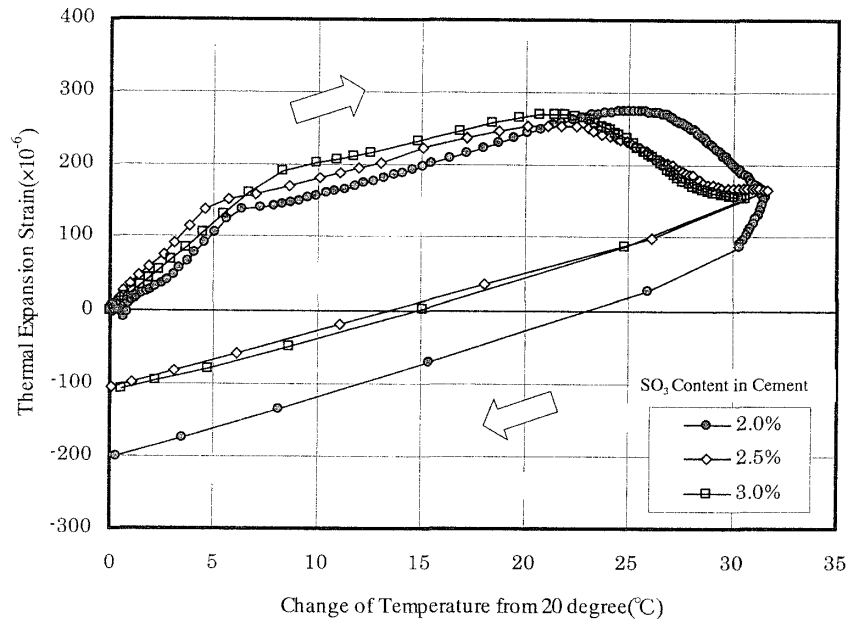(c) ; In case of low-heat portland blast-furnace slag cement

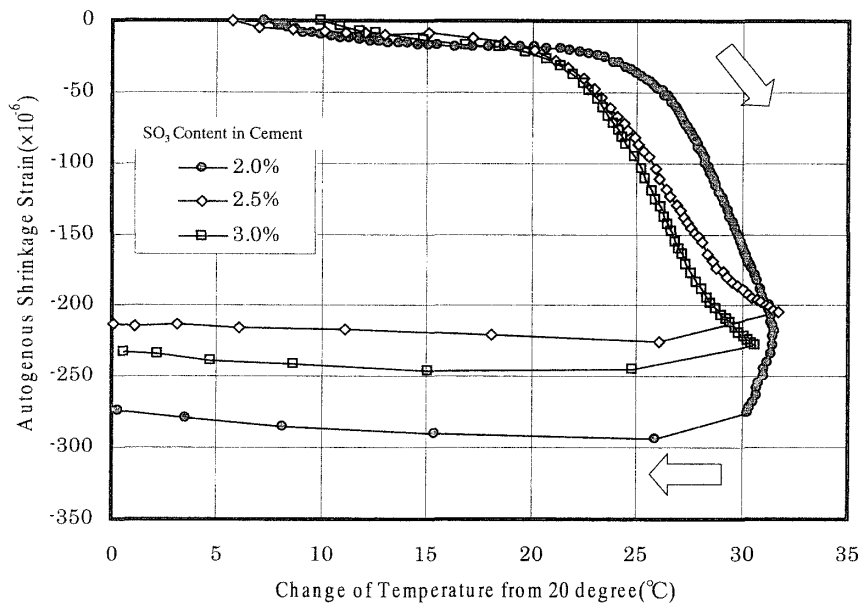**Fig.13** Relation between SO₃ content in cement and thermal expansion strain of concrete

Fig.14 Relation between SO₃ content in cement and autogenous shrinkage strain of concrete

percent showed rapid increase associated with the reversal thermal expansion strain, but the increase gradient was gentler than that of 2.0 percent and the autogenous shrinkage strain when the concrete temperature returned to 20°C was small. Thus an increase in SO₃ content in cement was proven to reduce the autogenous shrinkage strain of concrete under temperature histories.

(3) Effects of gypsum (SO₃) content in cement on the degree of hydration of blast-furnace slag (BS)

When SO₃ content in LB cement varied from 1.5 to 5.0 percent, degree of hydration of BS in LB cement (hereafter referred to as degree of hydration) at temperatures of 20 and 40°C were determined and shown in **Fig.15** and **Fig.16**. At a temperature of 20°C, there was almost no influence SO₃ content in cement affects degree of hydration until age of 3-days. However, degree of hydration showed the tendency increased, so that SO₃ content in cement decreased,
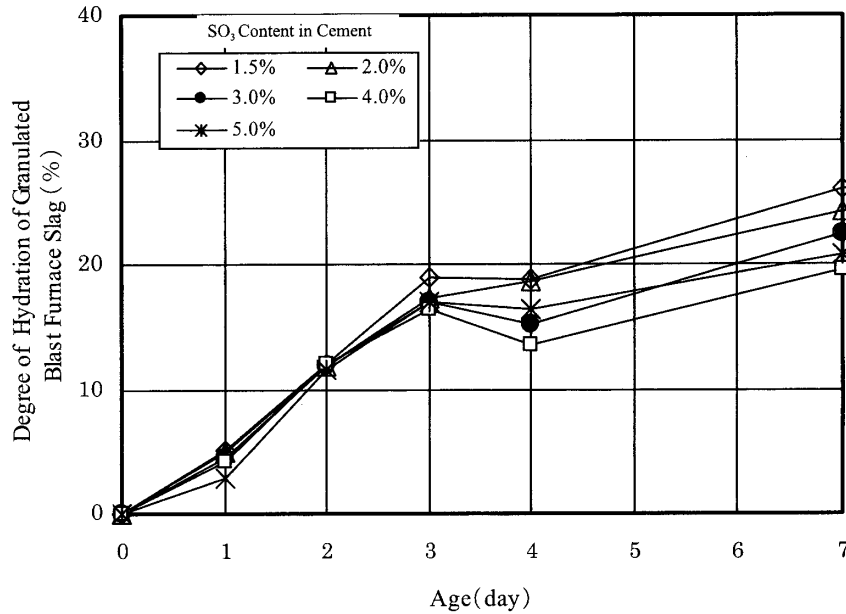
Fig.15 Relation between SO₃ content in LB cement and degree of hydration of blast-furnace slag in LB cement at a temperature of 20°C
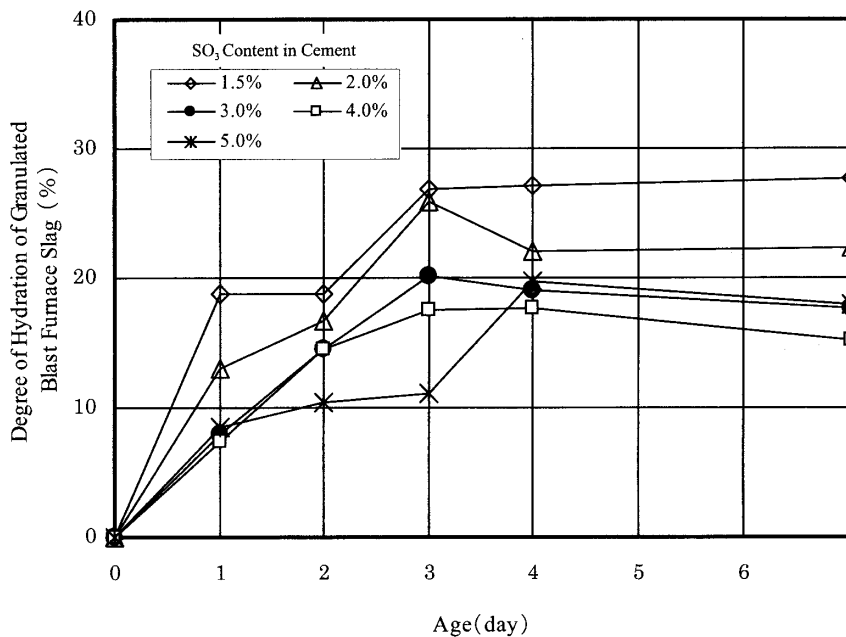

Fig.16 Relation between SO₃ content in LB cement and degree of hydration of blast-furnace slag in LB cement at a temperature of 40°C

when it passed age of 3-days. In case of a temperature of 40°C, degree of hydration went abruptly up in age of 3-days. However, the rapid rise of degree of hydration showed the tendency reduced as SO₃ content in cement was increased.

No remarkable difference in the degree of hydration at the age of 7-days was observed at a temperature of 20 and 40°C, while at early ages until 2-days, degree of hydration at a temperature of 40°C was greater than that at a temperature of 20°C. Thus the temperature dependency of BS hydration, i.e. reaction is activated at high temperatures, was confirmed.
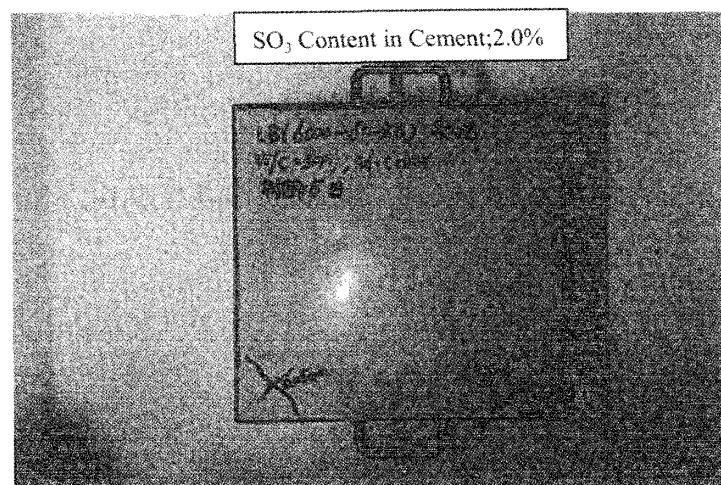
The hydration of BS in portland blast-furnace slag cement is activated at high temperatures and increases the amount of alumnum in the pore solution by loosening the silica-aluminum-oxygen chain structure that enclose calcium. As a result, excessive amount of
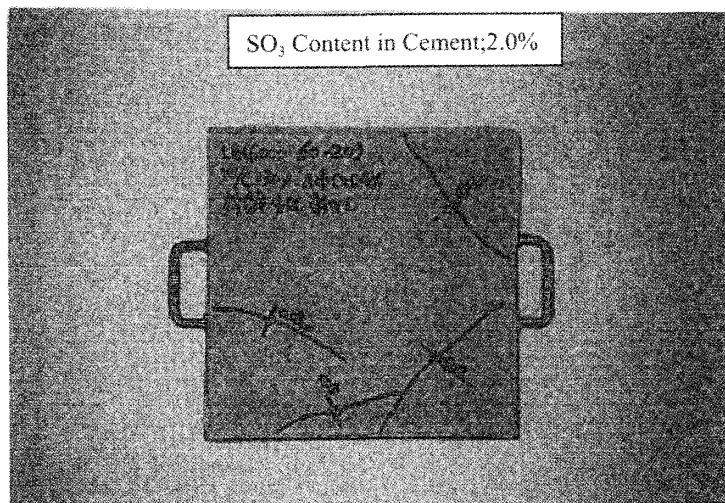
(Developed age 4-day,Maximum width 0.08mm)
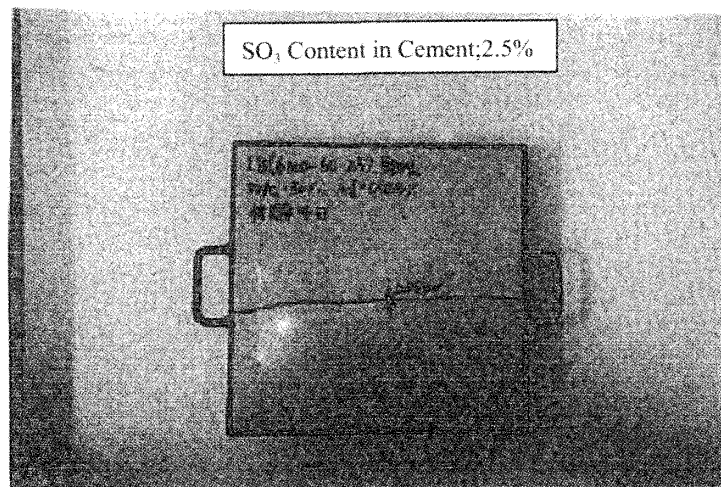
(Developed age 4-day,Maximum width 0.05mm)

(Developed age 5-dy,Maximum width 0.05mm)

Photo 1 Results of the observation of cracks

aluminum compared to that of sulfate ion, which is originated from the gypsum added to the cement, may accelerate the ettringite-monosulfate transition leading to an increase in autogenous shrinkage strain. A working mechanism of the addition of gypsum to reduce autogenous shrinkage is not limited to balance the sulfate ion against the cement-originated aluminum but also control abrupt supply of the slag-originated aluminum by inhibiting the temperature-induced slag hydration.

(4) Observation of cracks according to a change of gypsum (SO₃) content in cement

Cracks developing over cement pastes with an SO₃ content in LB cement of 2.0, 2.5 and 3.0 percent were visually inspected and are shown in **Photo 1**. No significant difference was observed in the crack width but the number and length decreased with an increase in SO₃ content in cement.

4. Concluding remarks

Low-heat portland blast-furnace slag cement undergoes considerable autogenous shrinkage strain at a temperature history, on which effects of gypsum phase and content in cement has been studied. Because aluminate hydrates are most affected by gypsum, the amount of transition of ettringite to monosulfate was determined for various types of portland blast-furnace slag cement. Major findings are as follows.

1) The autogenous shrinkage strain under temperature histories was determined under conditions of variable gypsum contents in cement, and the autogenous shrinkage strains at the maximum temperature and at the subsequent room temperature were reduced when gypsum (SO₃) content in the cement exceeded 2.5 percent. While no particular difference was observed between mixtures with gypsum (SO₃) content of 2.5 and 3.0 percent.

2) The abrupt hydration of granulated blast-furnace slag in cement at high temperatures was able to be reduced when the amount of gypsum (SO₃) content in cement was increased. Because the hydration of granulated blast-furnace slag releases aluminates that promote the transition from ettringite to monosulfate, addition of gypsum (SO₃) content in cement was supposed to control the hydration leading to a reduction of the autogenous shrinkage strain.

3) Crack developing of hardened cement pastes was observed to confirm the effect of gypsum (SO₃) content in cement. It was shown that the number and total length of crack decreased with an increase in SO₃ content, which was probably the major reason of reducing the autogenous shrinkage strain.

4) The aluminate hydrates in various type of hardened portland blast-furnace slag cement were analyzed by X-ray diffraction. It was shown that the transition from ettringite to monosulfate was recognized in each cement type. While the amount of ettringite transition was largest in low-heat portland blast-furnace slag cement that exhibited significant autogenous shrinkage and was a half of the low-heat blast-furnace slag cement in blast-furnace slag cement type B that exhibited lowest autogenous shrinkage strain. It was quantitatively shown that the reason was able to be attributed to the difference in molar volume of ettringite and monosulfate. Because the molar volume of monosulfate is less than about half of the ettringite, pores tended to shrink leading to an increase in autogenous shrinkage strain.

Thus through the study of the effect of gypsum in cement on the autogenous shrinkage, it was shown how the autogenous shrinkage is affected by the gypsum content in cement. Future tasks include practical method to reduce the autogenous shrinkage from the viewpoint

of a cement manufacturer.

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