



# Long-term volume changes and microcracks formation in high strength mortars

Shin-ichi Igarashi\*, Hiroshi R. Kubo, Mitsunori Kawamura

*Department of Civil Engineering, Kanazawa University, 2-40-20 Kodatsuno, Kanazawa 920-8667, Japan*

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## Abstract

Volume changes of high strength mortars cured in water were investigated. Effects of characteristic microstructure on the volume stability were considered in relation to the formation of microcracks. Mortars without silica fume exhibited swelling at early ages while the ones with silica fume started to swell at long ages after a certain period of initial shrinking in water. Cracking at long ages was confirmed for the both mortars with and without silica fume. However, there were distinct differences in the characteristics of crack patterns, such as the situation for cracking and their effects on the strength development between both. It was suggested that a mechanism other than autogenous shrinkage was involved in volume changes which occurred in mortars with an extremely low water/binder ratio. Generation of the internal expansive pressure due to the late cement hydration should be taken into account for a mechanism to cause microcracks in mortars at long ages. © 2000 Elsevier Science Ltd. All rights reserved.

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## 1. Introduction

High strength concretes with a low water/binder ratio are in general considered more durable than conventional concrete. High strength concrete has been often called high performance concrete. High performance in the high strength concrete results from dense microstructure characterized by a much refined pore system and a low porosity. Such a characteristic pore structure is supposed to greatly reduce volume changes of concrete.

On the other hand, high strength concrete with an extremely low water/binder ratio also has an intrinsic potential for volume changes. High strength concrete exhibits remarkable autogenous shrinkage at early ages. This contraction may cause cracks in premature concretes if the shrinkage is restrained so as to generate internal stresses beyond the tensile strength. Another different mechanism was also proposed for the volume change in the high strength concrete [1]. High strength concrete with an extremely low water/binder ratio contains a large amount of

unhydrated cement particles. Those unhydrated cement particles still have the potential for hydration. However, enough spaces for new hydration products are not present in the mature high strength concrete with a low porosity. Therefore, if the hydration of remnant cement particles takes place in the mature system, a volume increase must occur to provide the rooms for the new hydration products. As a result, cracks are induced by the development of the internal expansive pressure due to the accumulation of the products [1]. Actually, Odler et al. [2] investigated the volume changes of hardened cement pastes with an extremely low water/cement ratio. They reported that expansive strains due to the accumulation of the hydration products reached about 4–5%.

High strength concrete, therefore, has a different potential from conventional concrete in the volume changes derived from its characteristic microstructure [3]. Volume changes in concrete cause cracks so that high performances of high load bearing capacity and durability may be reduced. Therefore, it is important to investigate the effects of characteristic microstructures on the volume stability in high strength concrete.

The purpose of this study is to investigate the durability of high strength mortars stored in water. Effects of water/binder ratio and the addition of silica fume on the

\* Corresponding author. Tel.: +81-76-234-4621; fax: +81-76-234-4632.

E-mail address: igarashi@t.kanazawa-u.ac.jp (S. Igarashi).

volume stability of mortars are discussed by examining cracking in the mortars with the fluorescence microscope. Mechanisms of cracking at long ages are also discussed on the basis of the characteristics of crack patterns in the high strength mortars.

## 2. Experimental

### 2.1. Materials and mix proportion of concretes

Low water/binder ratio mortars with and without silica fume were produced. The cement was ordinary Portland cement. The fine aggregate was a natural river sand with F.M. of 2.49. A commercial silica fume with the specific surface area of 20.0 m<sup>2</sup>/g and SiO<sub>2</sub> content of 90.8% and a superplasticizer of the polycarboxylic acid type were used. For a comparison, normal strength mortar with a relatively high water/cement ratio of 0.55 was also prepared. Mix proportion of the mortars are given in Table 1.

### 2.2. Compressive strength test

Cylindrical specimens with 50 mm diameter and 100 mm height were prepared. The specimens were demolded and placed in water at 20°C at 24 h after casting. At the prescribed ages, compressive test was carried out according to JSCE G505-1995.

### 2.3. Flexural strength test

The 40 × 40 × 160 mm prisms were prepared and cured under the same condition as the specimens for compressive strength test. Flexural strength test of three-point bending was carried out according to JIS R 5201.

### 2.4. Shrinkage test

Mortar prisms of 40 × 40 × 160 mm with two gage plugs at its ends were prepared. The specimens were demolded at 24 h after casting. The initial length of mortar prisms was measured immediately after demolding. They

were cured in water at 20°C. Length changes of the specimens were measured by the contact gage method at prescribed times.

### 2.5. Fluorescence microscope examinations

At the prescribed ages, a slice with about 10 mm thickness was cut out from the middle of specimens for the shrinkage test. The slice was immersed in ethanol for at least 24 h, and then was impregnated with the epoxy resin containing a fluorescence dye. After the resin hardened at room temperature, the slice was polished with a SiC paper and diamond slurry.

## 3. Results

### 3.1. Compressive and flexural strength

Fig. 1 shows the compressive strength of mortars with and without silica fume. The compressive strength of mortars without silica fume increased with time although the gain in the strength was relatively small after 14 days (Fig. 1a). The strength of mortars with silica fume also increased with time up to 90 days (Fig. 1b). However, a slight reduction in the strength was observed for all the silica fume-containing mortars at 180 days. Differences in the strength between mortars with water/binder ratios of 0.24 and 0.30 were not so great in silica fume-containing mortars compared to those in the ones without silica fume.

Fig. 2 shows the flexural strength for mortars with and without silica fume at various ages. The trends are generally similar to those for the compressive strength. However, the reduction in flexural strength was found only for the silica fume paste at 180 days although the decrease in compressive strength was observed for all the specimens with silica fume (Fig. 1b).

It should be noted that the silica fume mortars exhibited lower strength than mortars without admixture at the same water/binder ratio. Namely, the addition of silica fume did not always result in the increase in strength at extremely low water/binder ratios.

Table 1  
Mix proportion of high strength mortars  
SP: superplasticizer.

Water/binder	Cement	Silica fume	Sand	SP (wt.% of binder)	Type of mortars
0.24	1	0	1	1.6	Portland cement mortar
0.24	0.9	0.1	1	4.0	Silica fume mortar
0.24	0.9	0.1	0	4.0	Silica fume paste
0.30	1	0	1	0.7	Portland cement mortar
0.30	0.9	0.1	1	1.0	Silica fume mortar
0.55	1	0	1.57	0	Portland cement mortar (volume fractions of components are adjusted)
0.55	0.9	0.1	1.54	0.5	Silica fume mortar (volume fractions of components are adjusted)

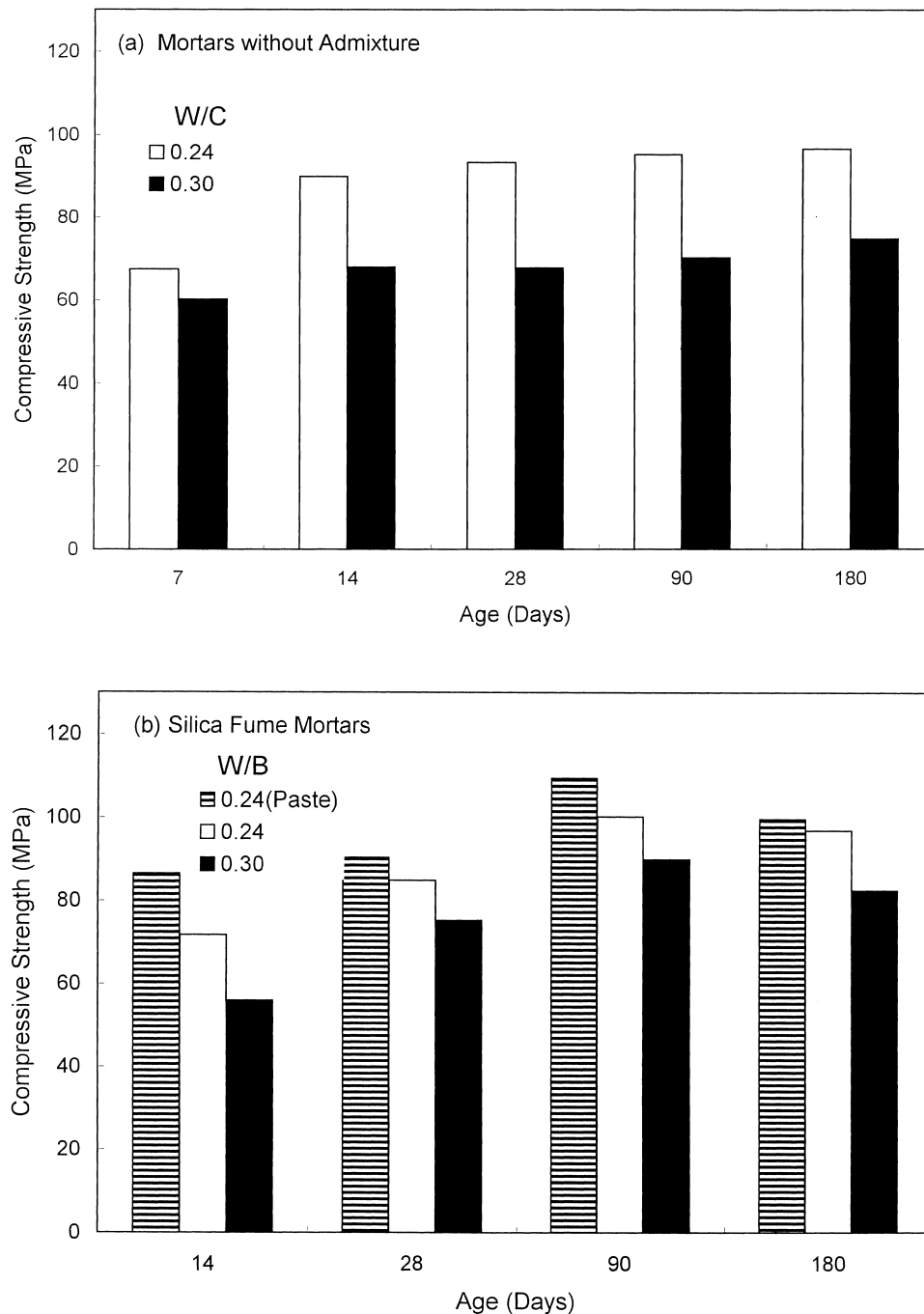


Fig. 1. Compressive strength of high strength mortars: (a) mortars without admixture and (b) silica fume mortars.

### 3.2. Length changes of mortars

Length changes in the mortars with and without silica fume are presented in Fig. 3. The mortars without silica fume swelled; the mortars with silica fume shrank. The mortars without admixture swelled at a great rate immediately after the immersion in water. The swelling appeared to have been stabilized at about 100 days. However, the swelling strain started increasing again at about 200 days. No difference was found in swelling strain between water/

cement ratio of 0.24 and 0.30 until at least the age of 250 days. Afterward, however, the mortars with a water/cement ratio of 0.24 swelled at a greater rate than the mortars with a water/cement ratio of 0.30.

The length change in concrete mainly depends on the volume change of hardened cement paste. In order to directly compare the swelling strains between low and high water/cement ratios, the mix proportions of both mortars are designed so that the volume fraction of cement paste in the mortar with a water/cement ratio of 0.55 was

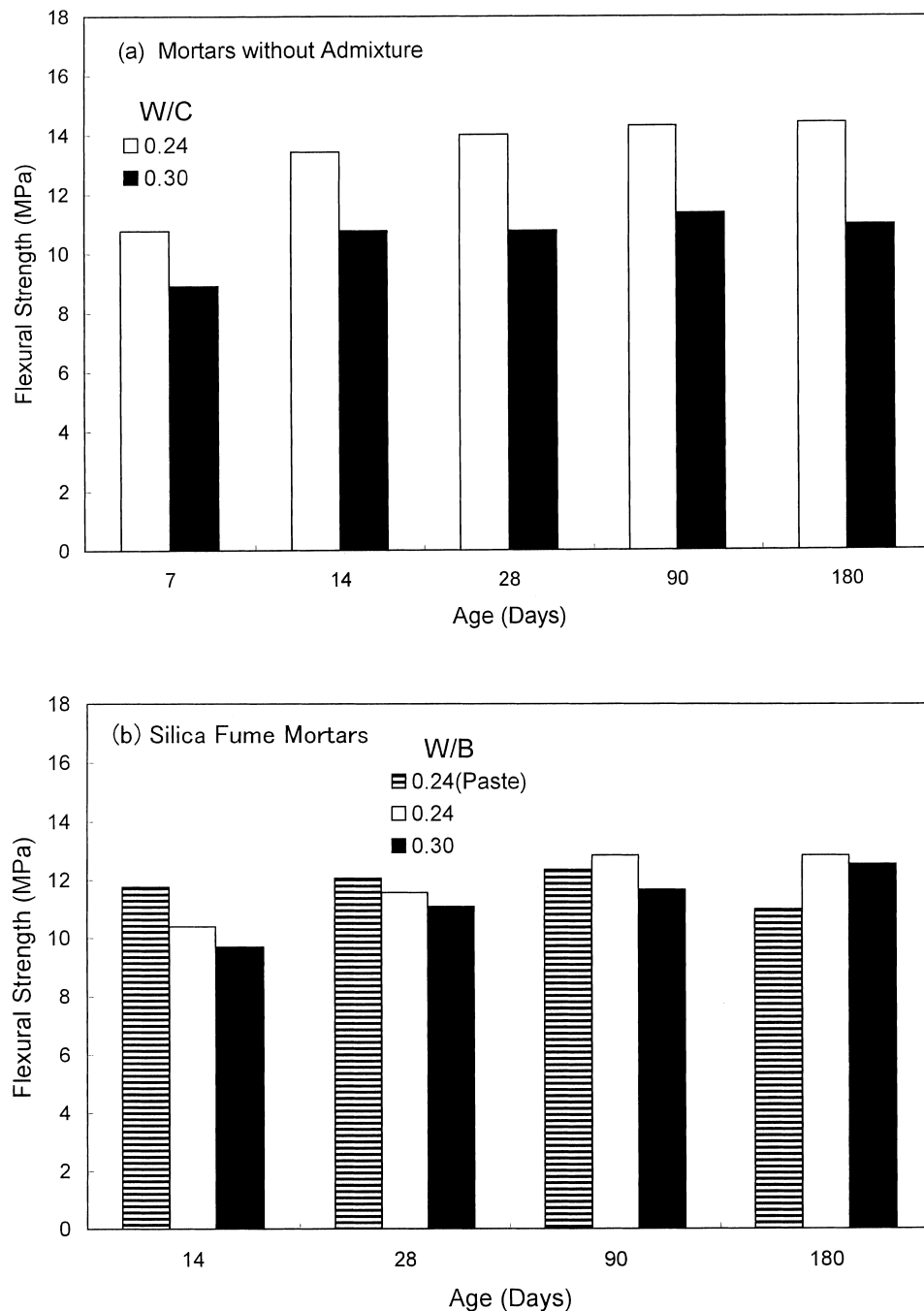


Fig. 2. Flexural strength of high strength mortars: (a) mortars without admixture and (b) silica fume mortars.

the same as in the mortar with a lower water/cement ratio of 0.24. The mortar with water/cement ratio of 0.55 as well as the other mortars with low water/cement ratios swelled rapidly during the first 50 days. However, the rate of swelling of mortars decreased with time. The swelling strains of mortars with a water/cement ratio of 0.55 were at around  $100\text{--}130 \times 10^{-6}$  after 100 days. These are half as small as in the mortar with lower water/cement ratios. Such relatively small strains in mortars with a higher water/cement ratio up to 100 days are comparable to those

for normal strength concrete. It is reported that the strain due to swelling was around  $100\text{--}150 \times 10^{-6}$  for conventional concrete [4].

Contrary to the swelling in mortars without admixture, silica fume mortars exhibited large shrinkage after they swelled to some extent during the first few days (Fig. 3b). The initial swelling and the subsequent shrinkage behavior depend on the water/binder ratio of mortars. The net shrinkage strain after the initial swelling at water/binder ratio of 0.24 reached about  $200 \times 10^{-6}$ ; that for mortars

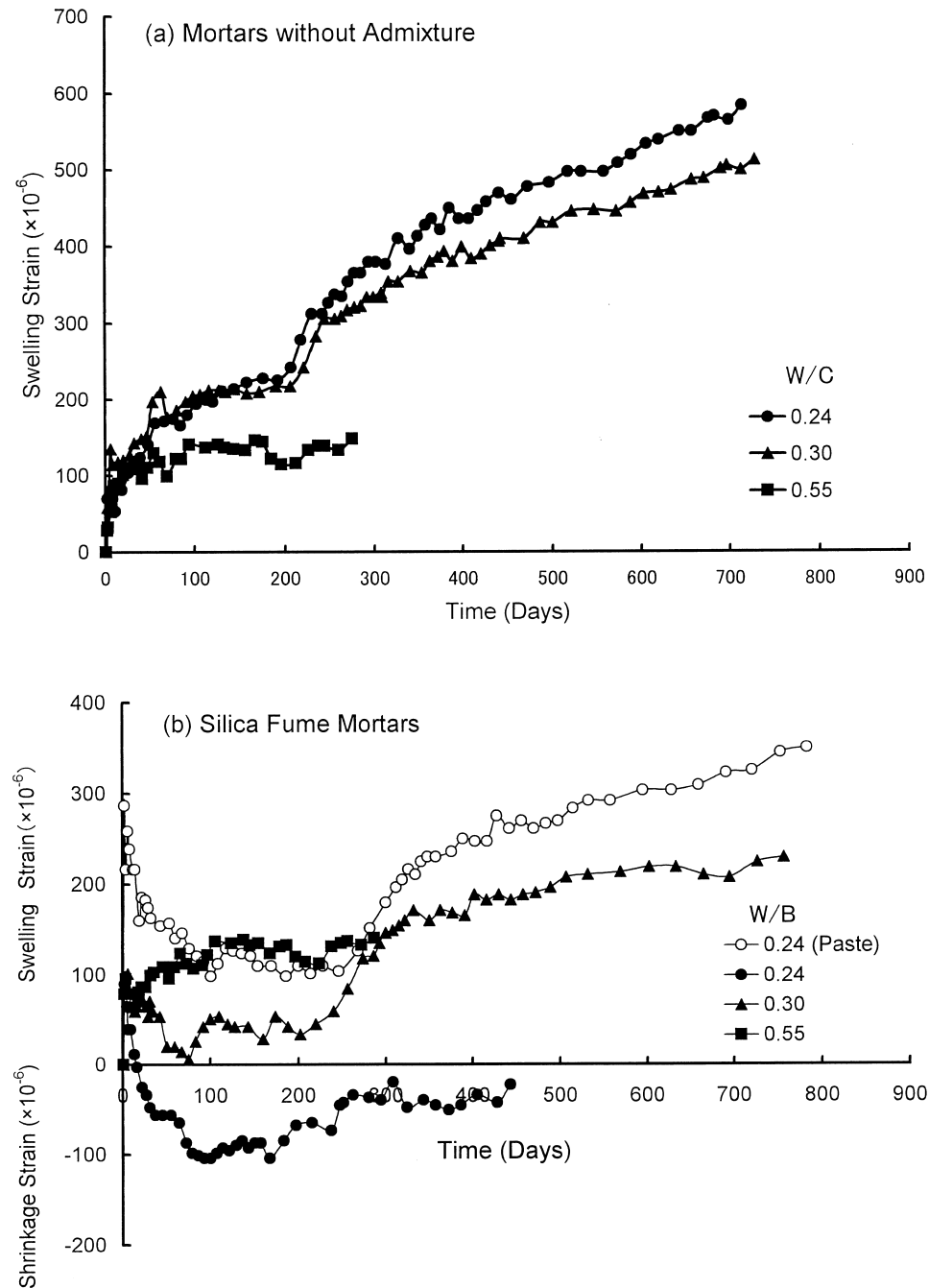


Fig. 3. Length changes in high strength mortars in water: (a) mortars without admixture and (b) silica fume mortars.

with a water/binder ratio of 0.30 about  $100 \times 10^{-6}$ . However, the silica fume mortars with water/binder ratio of 0.24 and 0.30 in water did not continue to shrink for a long time. The mortars started to swell at about 200 days after immersion in water. The silica fume paste also started to show a large swelling at almost the same time as the other two.

Such transition from shrinkage to swelling was not found in the silica fume mortar with a higher water/binder ratio of 0.55. The mortar with a high water/binder ratio has been continuing to swell immediately after immersion in

water. Its swelling strain was almost the same as that for the corresponding mortars without silica fume (Fig. 3a).

### 3.3. Cracking in mortars with low water/binder ratios

Fig. 4 shows the fluorescence micrographs for the polished surfaces of mortars without silica fume. At the age of 28 days, a few cracks along the periphery of aggregate grains were found (Fig. 4a). There were no characteristic cracks in the bulk cement paste phase for the initial few months. At a long age of 430 days, however, discrete short

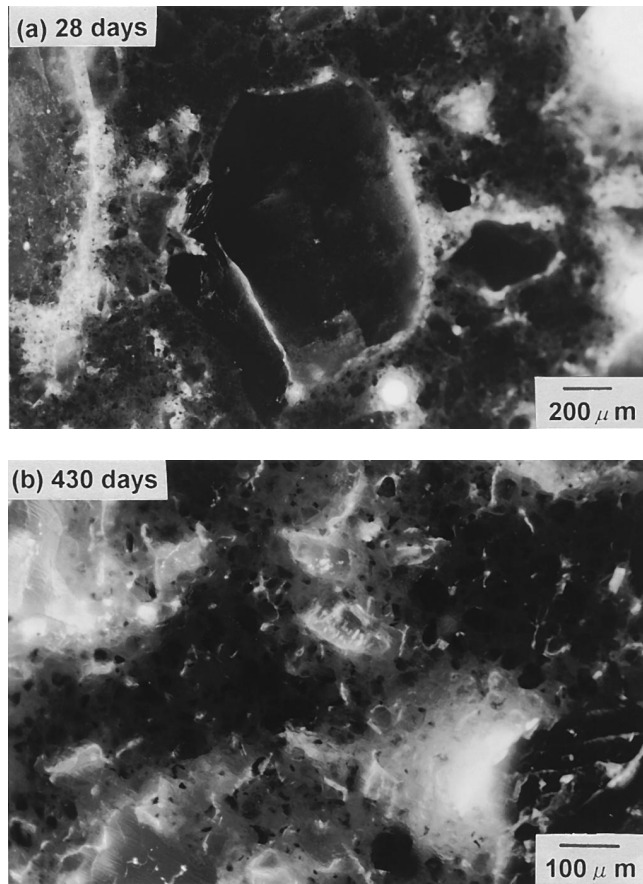


Fig. 4. Fluorescence micrographs for mortars without silica fume: (a) 28 days and (b) 430 days.

microcracks were dispersed all over the cement paste matrix, as shown in Fig. 4b. These discrete microcracks formed were directed towards random directions.

Fig. 5 shows the fluorescence micrograph for the polished section of silica fume mortar at the age of 170 days. Many cracks were found through the cement paste matrix. However, patterns of these cracks were clearly different from those in mortars without silica fume. Cracks in silica fume mortars were connected with one another forming networks. Their width was greater than those in mortars without silica fume. Furthermore, they seem to preferably develop in radial direction from surfaces of the aggregate. These characteristic patterns of cracking in silica fume mortars suggest the occurrence of relatively large volume changes in the cement paste matrix by the age of 170 days.

## 4. Discussions

### 4.1. Cracking mechanisms in mortars with and without silica fume

Cracking was confirmed in mortars with low water/binder ratios cured in water at long ages. Generally speak-

ing, cracking in cementitious composites is caused by volume changes of components if no mechanical forces are applied to the system. Indeed, all mortars at extremely low water/binder ratios in this study exhibited relatively great volume changes.

Cracks in the silica fume mortars are found to be formed during the period of shrinking of mortar specimens. As described above, cracks were developed to form continuous networks. On the contrary to such coalescence of cracks in silica fume mortars, cracks in mortars without silica fume were isolated. Furthermore, these cracks in mortars without silica fume were observed in the period of swelling. These differences in cracking characteristics suggest that the mechanisms of cracking in these mortars are different from each other.

#### 4.1.1. Mortars with silica fume

It is well recognized that the concrete with extremely low water/binder ratios exhibits considerable autogenous shrinkage. Autogenous shrinkage is generally more remarkable at early ages, especially during the initial period after the setting of cement. However, Persson [5] showed that the autogenous shrinkage continued for long period as far as self-desiccation lasted in high strength concrete. It is also pointed out that the autogenous shrinkage could not be fully prohibited even by curing in water [6,7]. Tazawa and Miyazawa [6] showed experimentally that the autogenous shrinkage in internal portions of specimens and the swelling at their nearby periphery occurred simultaneously in water curing, and the deformation of specimens as a whole was dependent on the size of specimens.

The coincidence of autogenous shrinkage and swelling results in the deformation gradient across the cross-section of specimen. It is supposed that the internal restraint due to the strain gradient may cause cracks [8]. However, such cracks caused by the non-uniform deformation must be oriented to specified directions. However, as shown in Fig. 5, the network of cracks radically developed from

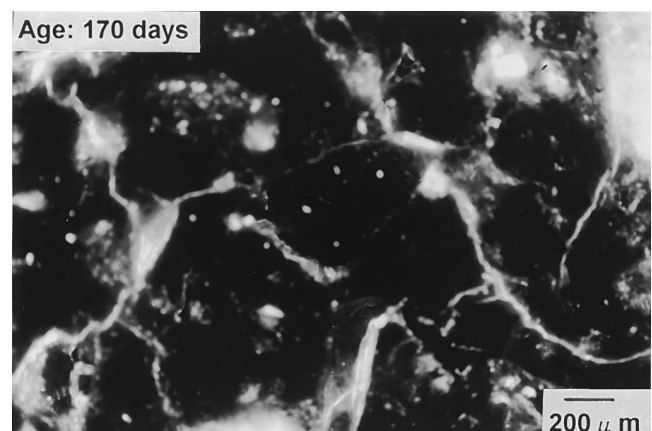


Fig. 5. Fluorescence micrograph for silica fume mortar (age = 170 days).

aggregate grains all over the cross-section. Therefore, the networks of cracks in this study cannot be due to the strain gradient across sections.

On the other hand, local stresses are induced even if autogenous shrinkage is isotropic. Namely, the autogenous shrinkage in the cement paste matrix also induces tensile stresses around aggregate particles. As a consequence, cracks would radiate from aggregate particles if the stresses are greater than the tensile strength [9]. The cracks shown in Fig. 5 seem to develop toward radial directions. Taking into account that these radial cracks were found during the period of shrinking, these cracks in silica fume mortars were caused by the restraint of autogenous shrinkage, by aggregate particles.

#### 4.1.2. Mortars without silica fume

It was also pointed out that the cement paste with an extremely low water/cement ratio had the potential for expansion after setting [1]. The expansion in the cement paste is related to its low porosity and the presence of many unhydrated cement grains. As mentioned earlier, Odler et al. [2] reported that the paste with a low porosity exhibited relatively great expansion, and the expansion strain was dependent not only on the hydration temperature, but also water/cement ratio. The expansion at a low water/cement ratio results from the accumulation of hydration products in the low porosity cement paste, which has only a limited pore space for new hydration products. Furthermore, Hillemerier and Schroder [1] revealed that the delayed hydration of unhydrated cement particles occurred at long ages by the diffusion of water. They

also showed that the internal pressure developed by the late hydration caused cracks leading to the reduction in the durability of high strength concrete.

As previously described, discrete microcracks were formed in admixture-free mortars with a water/cement ratio of 0.24 at least by 430 days. It is obviously demonstrated from Fig. 3 that discrete microcracks were formed in the admixture-free mortars during the period of the progress of active expansion. Furthermore, microcracks were dispersed within the bulk cement paste phase itself (Fig. 4b). The result of fluorescence microscope examinations appears to imply that aggregate particles in the mortars did not have anything to do with the formation of the microcracks in the paste. Taking into account the situation of cracking, the occurrence of microcracks in admixture-free mortars at long ages may be attributed to the development of internal pressure due to the late hydration of unhydrated cement grains.

Expansions for mortars with low water/cement ratios (Fig. 3) are much smaller than those for the paste with a low water/cement ratio obtained by Odler et al. [2]. However, it should be noted that the swelling strains for the mortars with low water/cement ratios in this study are much greater than those for a high water/cement ratio of 0.55. The mortars with a low water/cement ratio of 0.24 must have a smaller volume fraction of hydrated cement gel than mortars with a high water/cement ratio of 0.55 [10]. Therefore, much greater expansion in mortars with a low water/cement ratio implies that mechanisms other than the gel swelling are involved in great expansions in mortars with an extremely low water/cement ratio.

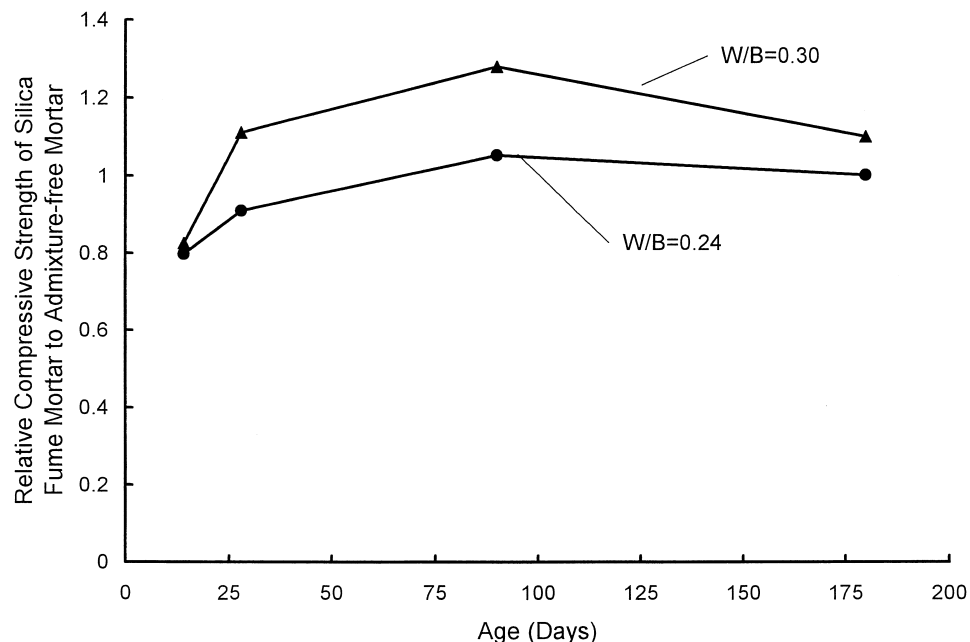


Fig. 6. Ratio of compressive strength of silica fume mortar to admixture-free mortar.

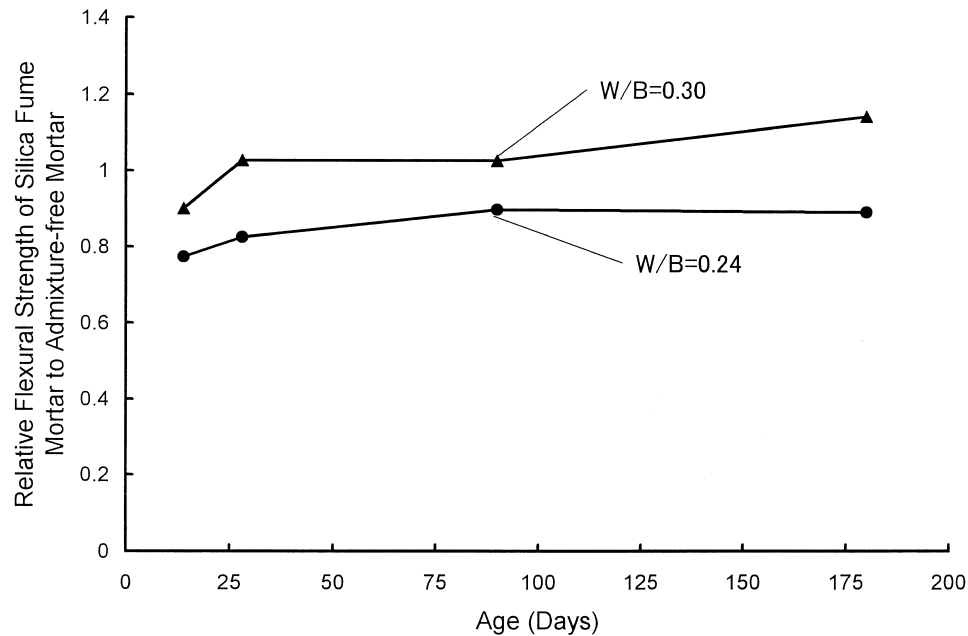


Fig. 7. Ratio of flexural strength of silica fume mortar to admixture-free mortar.

#### 4.2. Effect of cracking on the development of strength

It is of great interest whether the cracks formed at long ages have an adverse effect on the development of strength with time. As shown in Figs. 1a and 2a, the mortars without silica fume did not exhibit any reduction in strength until at least 180 days. Furthermore, cracks formed in the mortars at a long age were very fine. Cracks in mortars in this study were far smaller than those observed by Hillemerier and Schroder [1]. Cracks formed in this study were so fine that they had little effect on the reduction in strength in mortars.

Formation of continuous cracking networks in the whole cement paste phase, however, must have a serious effect on the strength in silica fume mortars. As presented in Fig. 1b, the strength of silica fume mortars decreased at 180 days. At that age, continuous crack networks were found by the fluorescence microscope examinations.

The ratio of compressive strength of silica fume mortars to that of admixture-free mortars is shown in Fig. 6. At a water/binder ratio of 0.30, the strength of silica fume mortars was a little greater than that of admixture-free mortars. However, at a lower water/binder ratio of 0.24, silica fume mortars had lower strength than admixture-free mortars. Namely, the addition of silica fume did not increase the strength of mortar at an extremely low water/binder ratio of 0.24.

Fig. 7 shows the time-dependent changes of the ratio of flexural strength of silica fume mortars to that of admixture-free mortars. The ratios for flexural strength are lower than those for the corresponding compressive strength. Namely, the flexural strength of mortars was considerably reduced by the addition of silica fume at a lower water/binder ratio of

0.24. The flexural strength is more sensitive to the formation of microcracks than the compressive strength. Therefore, the flexural strength in silica fume mortars at a water/binder ratio of 0.24 was reduced by the internal restrained shrinkage cracking.

#### 5. Conclusions

Volume changes of mortars with extremely low water/binder ratios were investigated. Mechanisms for expansion and shrinkage in water curing were discussed in relation to characteristic microstructure formed at extremely low water/binder ratios. Major results obtained in this study are summarized as follows:

1. Mortars without silica fume exhibited continuous expansion when cured in water. Discrete microcracks were found in the bulk cement paste phase at long ages.
2. Mortars with silica fume changed its behavior from shrinking to expanding in water. Continuous crack networks were found during the period of initial shrinkage.
3. When silica fume mortars with a low water/binder ratio were cured in water, shrinkage and swelling occurred simultaneously. However, the volume change at extremely low water/binder ratio results from the presence of silica fume.
4. Crack networks in silica fume mortars were induced by the internal restraints to autogenous shrinkage.
5. Expansive pressure generated by the late cement hydration in mortars without admixture should be



considered as a mechanism of the formation of discrete microcracks at long ages.

6. Formation of continuous crack networks due to autogenous shrinkage led to the reduction in strength of high strength mortars with silica fume. However, discrete microcracks caused by the late cement hydration had little effect on the development of strength in admixture-free mortars.

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