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# Influence of Microstructural Change under Stress on the Strength-Related Properties of Hardened Cement Mortar and Paste

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*Microstructural change of hardened cement paste and mortar under various stresses was studied to obtain the basic data for judging the safety of concrete structure repeatedly receiving the stress. The crack revealed generally at 60% of the fracture stress, and it grew rapidly, exceeding at 80% of the fracture stress. The growth of the cracks in the hardened body was more conspicuous in paste than in mortar, in fly ash cement than in normal portland cement and blastfurnace slag cement, at high W/C than at low W/C, and in repeated loading than in single loading. Results were discussed in connection with the changes of pore structure and backscattered electron images of hardened bodies after loading the stress.* ADVANCED CEMENT BASED MATERIALS 1997, 6, 87–98. © *1997 Elsevier Science Ltd.*
**KEY WORDS:** Compressive stress, Fracture stress, Repeated stress, Velocity of ultrasonic pulse propagation, Pore structure, Microstructure, Transition zone, Hardened mortar, Hardened cement paste, Normal portland cement, Blastfurnace slag cement, Fly ash cement

Since the compressive strength applied to such a heterogeneous composite material as mortar and concrete containing pores is concentrated in peculiar points including pores in the material, cracks develop and propagate in it, resulting in a collapse. Therefore, it is estimated that the single or repeated or continuous loading of stress affects the texture and structure of a hardened body.

Although many papers [1,2] have reported on the fatigue and fracture mechanism of concrete, few papers were found dealing with the microstructural change of concrete under stress.

The changes of the compositions and microstructures of hardened bodies of normal portland cement, blastfurnace slag cement (slag content: 50%), and fly ash cement (fly ash content: 25%) prepared at the water/cement ratio (W/C) of 0.65 and 0.4 before and after loading various types of stress were studied using measurement of the velocity of propagation of supersonic wave, pore size distribution, and backscattered electron image aimed at obtaining the basic data for judging the safety of concrete repeatedly and continuously receiving the stress caused by earthquake and heavy traffic and investigating the relationship between structural change and compressive stress in the hardened bodies until collapse.

## Experimental

### *Sample and Test Specimen*

Normal portland cement, blastfurnace slag cement (slag content: 50%), fly ash cement (fly ash content: 25%), and the standard sand from Toyoura listed in Table 1 were used for the experiment. Two mixture proportions were applied according to JIS mix (W/C = 0.65 and S/C = 2.0) and at W/C of 0.4 and S/C of 1.0, and two mixture proportions for cement paste were applied at W/C of 0.65 and 0.4. Since approximately 5% of mixing water was separated until hardening by bleeding in the cement paste prepared at W/C of 0.65, the actual W/C of the cement paste is considered to be 0.62. For making the flow values of the mortar and cement paste prepared at W/C of 0.4 equal to those prepared at W/C of 0.65, 2.0% of naphthalenesulfonic acid-based, high performance water-reducing agent to the amount of cement was added to those mixtures. The test specimens were prepared by curing in relative humidity of 95% at 20°C for 1 day, demolding from the molds, and then further curing in water until the age of 28 days.

### *Measurement of Compressive Strength and Velocity of Propagation of Supersonic Wave*

The compressive strengths of cement paste and mortar were measured according to JIS R 5201 using the

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**TABLE 1.** Chemical composition and fineness of normal portland cement, blastfurnace slag cement, fly ash cement, and sand

| | Chemical Composition (%) | | | | | | | | Bl' |
|---|---|---|---|---|---|---|---|---|---|
| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $Na_2O$ | $K_2O$ | ($cm^2/g$) |
| Normal portland cement (NPC) | 21.6 | 5.3 | 3.3 | 64.9 | 1.1 | 2.3 | 0.45 | 0.35 | 3,310 |
| Type B—blastfurnace slage cement (BSC) | 25.9 | 8.6 | 1.8 | 55.9 | 4.1 | 2.2 | 0.27 | 0.36 | 3,710 |
| Type C—fly ash blended cement (FAC) | 31.3 | 8.5 | 5.0 | 49.8 | 1.2 | 2.0 | 0.51 | 0.49 | 3,340 |

| | Residue (%) | | | | Volume Weight |
|---|---|---|---|---|---|
| | 300 μm | 212 μm | 150 μm | 106 μm | (kg/l) |
| Standard sand of JIS (Toyoura) | 0.1 | 46.2 | 95.8 | 99.5 | 1.52 |

*Note*: Bl' Blaine specific surface area.

sample prepared by molding and curing at 20°C until the age of 28 days. The velocity of propagation of supersonic wave was measured with a supersonic wave type quality tester of concrete (Pundit) manufactured by C.N.S. Electronics Ltd. (London).

### *Mortar for Single and Repeated Loading of Stress to Test Specimen*

The loading test of stress was made using test specimens cured for 28 days. Each single loading test of stress was made by increasing the compressive stress up to 30 MPa at intervals of 10 MPa and exceeding 30 MPa at intervals of 5 MPa. The loading time at the maximum stress was 1 minute. The fracture stress is the stress corresponding to the compressive strength.

The repeated loading stress test was made by loading 1,000 and 10,000 times a sine wave-formed partial pulsating stress corresponding to 15% to 60% of the fracture stress to a cubic test specimen of 4 cm edge cut out from the above-mentioned test specimen with an electrohydraulic servo fatigue tester (Instron 1331 type). The frequency of loading of stress is 5 cycles/s (Hz).

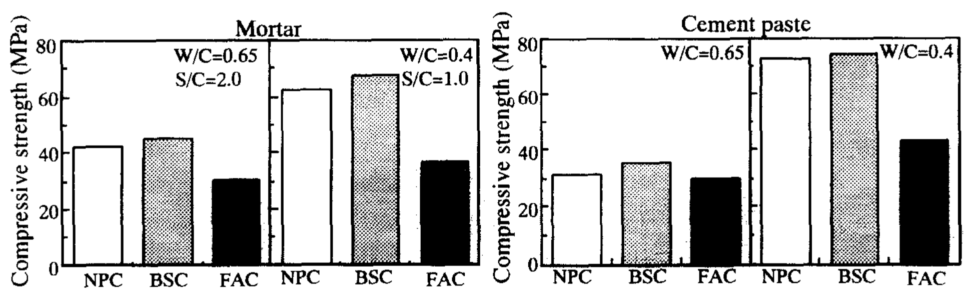
### *Analyses of Texture and Microstructure of Test Specimen*

After cutting out cubic samples of about 5 mm edge from hardened cement paste and mortar before and after the loading of stress using a diamond cutter, the hydration was terminated with acetone. The samples were D-dried and submitted to measurement of pore size distribution and observation of backscattered electron image. Pore size distribution was measured with a mercury-intrusion porosimeter, and texture and microstructure were observed by the backscattered electron images obtained by electron probe micro-analyzer (EPMA). Hardened bodies before loading of stress were submitted to the same measurement as those for comparison as needed.

## Results and Discussion

### *Physical Properties of Hardened Mortar and Hardened Cement Paste*

COMPRESSIVE STRENGTH. As shown in Figure 1, the compressive strengths at the age of 28 days of JIS mortar and mortar prepared at W/C of 0.4 from normal portland cement were 41.3 and 62.7 MPa, respectively, and those of cement paste prepared at W/C of 0.65 and W/C of 0.4 were 32.6 and 73.6 MPa, respectively. Although the compressive strength of cement paste prepared at W/C of 0.65 was lower than that of mortar because of conspicuous bleeding, that of cement paste prepared at W/C of 0.4 was approximately 20% higher than that of mortar. The compressive strengths at the age of 28 days of JIS mortar and mortar prepared at




NPC : Normal portland cement, BSC: 50% blastfurnace slag blended cement, FAC: 25% fly ash blended cement

**FIGURE 1.** Compressive strength of mortar and cement paste (20°C, 28 days).

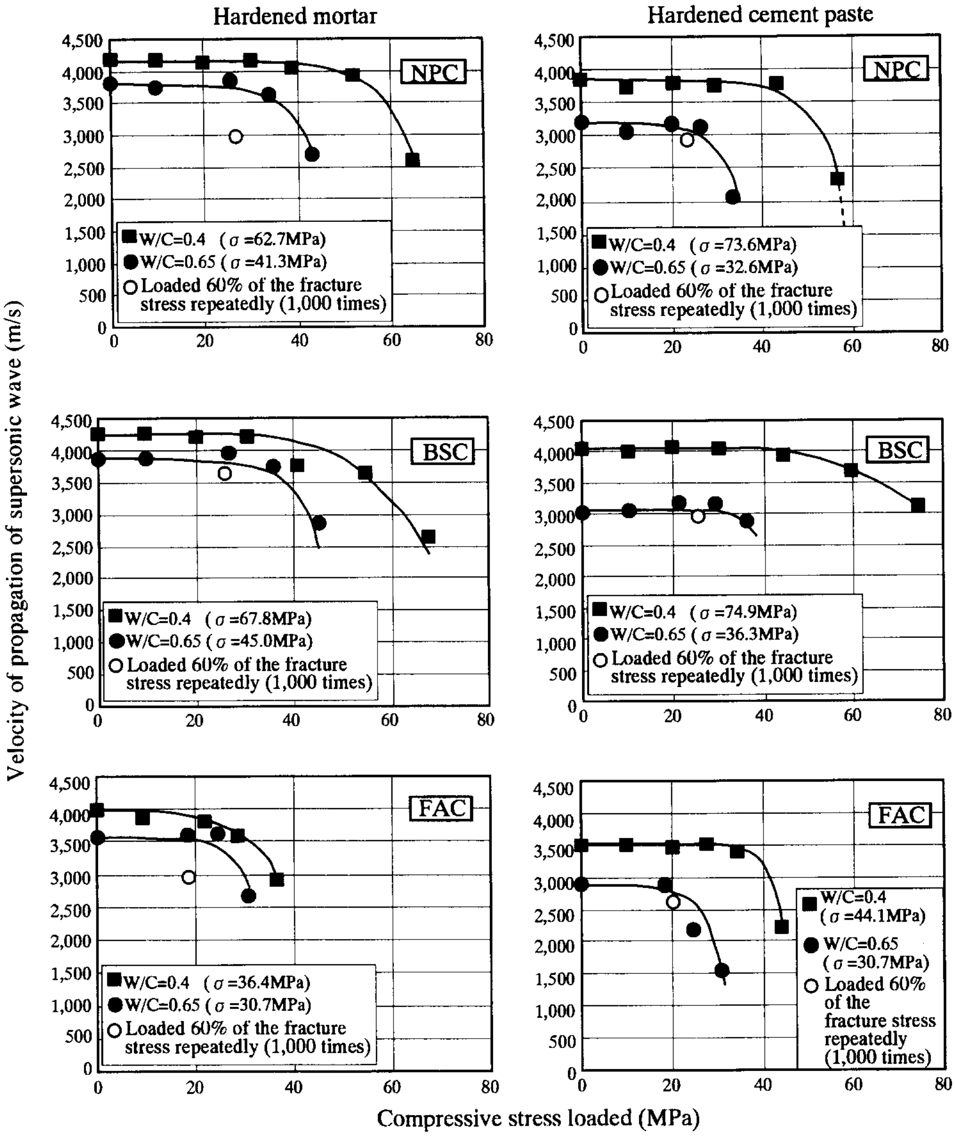



**FIGURE 2.** Relationship between compressive stress loaded and velocity of propagation of supersonic wave in hardened mortar and cement paste.

W/C of 0.4 from blastfurnace slag cement were 45.0 and 67.8 MPa, respectively, and those of cement paste prepared at W/C of 0.4 and W/C of 0.65 were 36.3 and 74.9 MPa, respectively. The compressive strengths of mortar and cement paste prepared from blastfurnace slag cement except the cement paste prepared at W/C of 0.4 were approximately 10% higher than those prepared from normal portland cement. The compressive strengths at the age of 28 days of JIS mortar and mortar prepared at W/C of 0.4 from fly ash cement were 30.7 and 36.4 MPa, respectively, and those of cement paste prepared at W/C of 0.65 and 0.4 were 30.7 and 44.1 MPa, respectively. The compressive strength of mortar and cement paste prepared from fly ash cement except the cement paste prepared at W/C of 0.65 were 30% to 40% lower than those prepared from normal portland cement. The strengths of mortar and cement paste prepared from fly ash cement were developed little within the age of 28 days even when the W/C was lowered to 0.40.

#### VELOCITY OF PROPAGATION OF SUPERSONIC WAVE

*Single Loading of Stress.* The variation of the velocity of propagation of supersonic wave in hardened mortar and cement paste after loading the compressive stress is illustrated in Figure 2.

The higher the density of a sample, the higher the velocity of propagation of supersonic wave generally was. The velocities of propagation of supersonic wave in hardened mortar prepared at W/C of 0.65 and 0.4 before loading the stress were 3920 and 4290 m/s, respectively. Although the latter was prepared using a small quantity of higher-density aggregate than cement paste, it was higher than that in the former. Possibly

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this is because the texture of hardened cement paste composing the matrix is denser in the latter than in the former.

The velocity of propagation of supersonic wave in normal portland cement mortar decreased sharply when the compressive stress exceeded 80% of the fracture stress. Those in mortar prepared at W/C of 0.65 and 0.4 went down to 2700 and 2600 m/s, respectively, by loading the fracture stress, whereupon those values were 31% to 39% lower than those before loading the stress. This suggests that the hardened texture of mortar is changed when loaded compressive stress reaches approximately 80% of the fracture stress.

The velocity of propagation of supersonic wave in blastfurnace slag cement mortar was decreased less by loading the stress than that in normal portland cement mortar. This suggests that large cracks impeding the propagation of supersonic wave are less formed in the former than in the latter.

The velocity of propagation of supersonic wave in fly ash cement mortar was hardly decreased by loading the stress at approximately 60% of the fracture stress in the same manner as in normal portland cement mortar. It began decreasing by loading the stress at approximately 80% or more of the fracture stress and sharply decreasing by loading the stress approaching the fracture stress.

The velocities of propagation of supersonic wave in normal portland cement paste before loading the stress prepared at W/C of 0.65 and 0.4 were 3200 and 3880 m/s, respectively. These values are lower than those in mortar containing aggregate, which suggests that the texture of hardened cement paste is porous. The velocity of propagation of supersonic wave in hardened cement paste began decreasing when the stress loaded exceeded approximately 60% of the fracture stress and sharply decreased when the stress exceeded approximately 80% of the fracture stress in the same manner as in mortar. The degree of decrease of the velocity of propagation of supersonic wave was higher than that in mortar. The velocity of propagation of supersonic wave in hardened cement paste prepared at W/C of 0.65 was reduced by loading the fracture stress to as low as 2100 m/s, which was approximately 34% lower than that before loading the stress. The velocity of propagation of supersonic wave in the hardened cement paste prepared at W/C of 0.4 was impossible to measure because the test specimen was completely collapsed by loading the fracture stress. This suggests that the fracture toughness of hardened cement paste is lower than that of hardened mortar.

The velocity of propagation of supersonic wave in blastfurnace slag cement paste was higher than in the other types of cement paste. This suggests its texture is denser than that of the latter. The velocity of propagation of supersonic wave in blastfurnace slag cement began decreasing by loading the stress at 60% of the fracture stress at a lower rate than in normal portland cement paste and fly ash cement paste. Those values in blastfurnace slag cement paste prepared at W/C of 0.65 and 0.4 when the fracture stress was loaded were 2900 and 3200 m/s, respectively, which were 90% and 80% of the values before loading the stress, respectively. These values were higher than the 2100 m/s (66%) in normal portland cement paste prepared at W/C of 0.65 and 1500 m/s (50%) and 2250 m/s (65%) in fly ash cement paste prepared at W/C of 0.65 and 0.4. The velocity of propagation of supersonic wave in fly ash cement paste before the loading of stress was lower than that in the other types of cement paste. This suggests that the texture is rougher. The velocity of propagation of supersonic wave in fly ash cement paste was sharply decreased by loading the stress exceeding 80% of the fracture stress in the same manner as the other two types of cement paste.

*Repeated Loading of Stress.* When the stress at 60% of the fracture stress according to the respective types of cement was applied repeatedly 1000 times, the velocities of propagation of supersonic wave in normal portland cement mortar, blastfurnace slag cement mortar, and fly ash cement mortar prepared at W/C of 0.65 were 3000, 3600, and 2950 m/s, respectively, which were lower by 20%, 5%, and 18%, respectively, than those obtained by single loading of stress. Each hardened mortar was collapsed by repeatedly loading the stress approximately 4000 times. Every test specimen prepared at W/C of 0.4 was collapsed by repeated loading of stress 200 times. Possibly this is because the stress loaded is easily concentrated in specified places in the hardened texture of mortar than that of relatively homogeneous cement paste, which results in easy destruction of the hardened texture by repeated loading of stress.

Hardened cement paste prepared at W/C of 0.65 was not collapsed even by repeating the loading of stress as many as 10,000 times. The velocities of propagation of supersonic wave in any type of cement paste after repeatedly loading the stress at 60% of the fracture stress 1,000 and 10,000 times were almost the same as those after the single loading of stress at 60% of the fracture stress. On the contrary, any type of cement paste prepared at W/C of 0.4 was collapsed by repeated loading of stress approximately 300 times. Possibly this is because the brittleness and Young's modulus are increased by the development of high strength, thereby facilitating the propagation of cracking.

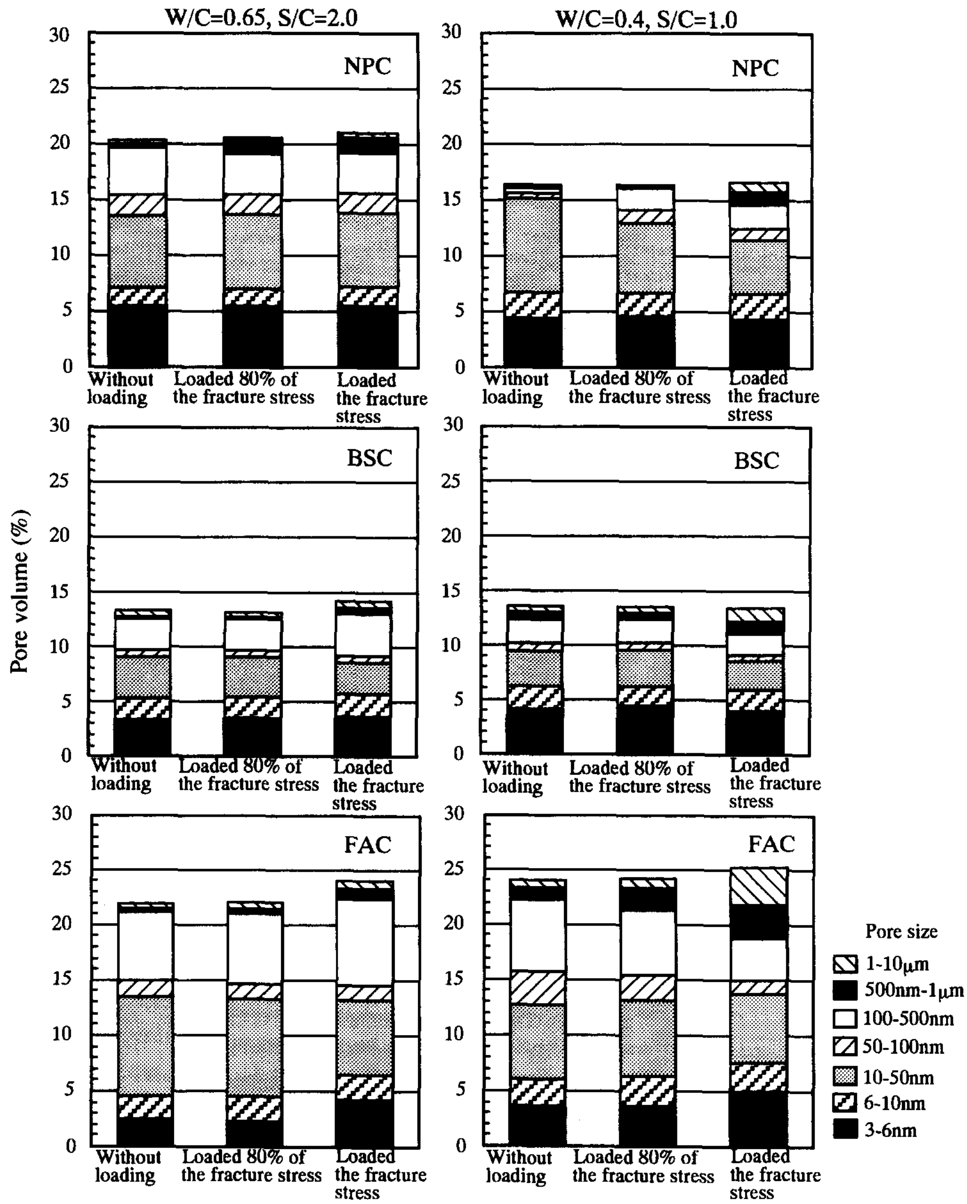



**FIGURE 3.** Relationship between compressive stress loaded and pore size distribution of hardened mortar.

## *Pore Structures of Hardened Mortar and Cement Paste*

**PORE STRUCTURE OF HARDENED MORTAR AFTER SINGLE LOADING OF STRESS.** The changes of the total pore volume and pore size distribution in mortar by loading the compressive stress are illustrated in Figure 3.

Mercury porosimetry is used widely due to its simple operation and high accuracy compared with the $N_2$ adsorption-desorption method [3], although there are some problems as to whether or not the pore space [4] type model shows the real-shaped pore space, mercury is pressed into the bottle-shaped pore space because the pressure of mercury is limited by the diameter of exit [4], and the sample is deformed by compression [4,5].

The total pore volume in mortar was increased little by loading the compressive stress at 80% or less of the fracture stress to mortar. However, when loading the stress exceeding 80% of the fracture stress, the large diameter pores and the total pore volume were increased, especially by loading the fracture stress.

The increase of the total pore volume in normal portland cement mortar prepared at W/C of 0.65 by loading the fracture stress is attributed to the volume increase of pores as large as 500 nm to 10 μm in diameter. Although the volume of pores 50 to 100 nm in diameter was reduced from 2.5% to 2.1% and that 100 to 500 nm in diameter was reduced from 4.3% to 3.8% by loading the fracture stress, that of pores 50 nm or less in diameter was hardly changed. This suggests that capillary pores as large as 50 to 500 nm in diameter mainly existing in the transition zone [6,7] around the aggregate are broken into pores as large as 500 nm to 10 μm in diameter by loading the stress with a specified magnitude or more. The pore volume 50 to 100 nm in diameter was reduced a little, while that 500 nm to 1 μm in diameter was increased by the loading of stress at 80% of the fracture stress. From this it is inferred that the destruction of the texture of mortar extends to the peripheral sections of pores 50 to 100 nm in diameter, whereas the diameter of newly formed pores is enlarged by the connection of pores 50 to 100 nm in diameter.

The increase of the total pore volume in mortar prepared at W/C of 0.4 is attributed mainly to the increase of the pores 100 nm to 10 μm in diameter. These diameters are slightly smaller than in mortar prepared at W/C of 0.65. The pore volume 10 to 50 nm in diameter was reduced from 8.4% to 4.6% by loading the stress, while those of 100 to 500 nm, 500 nm to 1 μm, and 1 to 10 μm in diameter were increased from 0.6% to 2.2%, 0.1% to 1.1%, and 0.2% to 1.0%, respectively. When loading the stress at 80% of the fracture stress, the pores 10 to 50 nm in diameter were reduced but the pores 100 to 500 nm and 500 nm to 1 μm in diameter were remarkably increased. Since few capillary pores as large as 50 nm or more in diameter are contained in mortar prepared at W/C of 0.4 compared with mortar prepared at W/C of 0.65, a relatively large number of slightly smaller pores 10 to 50 nm in diameter existing in the former are considered to be broken first by loading the stress into pores 100 nm or more in diameter. Since the pores 10 nm or less in diameter were hardly changed, these pores are considered to be not broken.

The total pore volume was hardly increased but the pores 10 to 50 nm in diameter were broken by loading the stress, thereby increasing the pores 50 nm or more in diameter in blastfurnace slag cement mortar prepared at W/C of 0.65. The trend is similar to that of normal portland cement mortar prepared at W/C of 0.4. Loading the fracture stress to blastfurnace slag cement mortar prepared at W/C of 0.4, the total pore volume was hardly increased and the pores as small as 10 to 50 nm in diameter were decreased, thereby increasing those 500 nm or more in diameter. Loading the stress at 80% of the fracture stress, the pore size distribution was hardly changed though the pores 10 to 50 nm in diameter were reduced a little. This agrees with the trend that the velocity of propagation of supersonic wave in this mortar is hardly reduced. Possibly this is because C-S-H is produced by the pozzolanic reaction of the slag with $Ca(OH)_2$ consuming it in blastfurnace slag cement mortar, and the texture is densified so much that the total pore volume and capillary pores 50 nm or more in diameter are reduced, and because the stress required for collapse of hardened body is increased, accompanied by the increase of strength of it, whereupon the collapse proceeds to even small diameter pores.

Loading the stress at 80% of the fracture stress to fly ash cement mortar prepared at W/C of 0.65, the pores 6 to 100 nm in diameter were slightly decreased and the pores 6 to 500 nm in diameter were decreased at W/C of 0.4. At both cases, the pores 500 nm or over in diameter were increased. Loading the fracture stress to the mortar prepared at W/C of 0.65 and 0.4, the total pore volume and the pores 3 to 6 nm and 100 nm or more in diameter were increased, and the pores 10 to 50 nm and 50 to 500 nm in diameter were decreased in the mortar prepared at W/C of 0.65 and 0.4, respectively. This is because the texture of transition zone at the interface of aggregate and cement paste and relatively rough texture in paste matrix are mainly broken into larger pores by loading the stress. The volume of pores 3 to 6 nm in diameter was increased, though that of pores 6 to 50 nm in diameter was not changed. From this it is inferred that not only do the pores with intermediate diameters develop into large cracks by loading the fracture stress, but new fine cracks also develop.

**PORE STRUCTURE OF HARDENED CEMENT PASTE AFTER SINGLE LOADING OF STRESS.** The cumulative pore volume of normal portland cement paste subjected to fracture stress and the difference in the cumulative pore volume of various types of cement paste before and after the loading of the fracture stress are illustrated in Figures 4 and 5.

Although the difference of pore size distributions in the range from 200 nm to 100 μm in normal portland cement paste prepared at W/C of 0.65 before and after the loading of the fracture stress was hardly observed, the pores 50 to 200 nm in diameter were increased remarkably by the loading of stress. The volume of pores 10 nm or less in diameter is hardly changed even by loading the fracture stress; accordingly, those pores were not broken by it. Although the volume of the pores 10 nm to 10 μm in diameter, particularly 10 to 100 nm in diameter, in normal portland cement paste prepared at W/C of 0.4 was increased by the loading of the fracture stress, the difference of the volume of the pores 10 nm or less in diameter before and after the loading of the fracture stress was reduced. The pores 10 nm in diameter or less is considered to be broken into pores 10 to 100 nm in diameter. Compared with the fracture stress to normal portland cement paste prepared at W/C of 0.65, it is doubled, whereupon the fracture propagated the texture in the vicinity of pores 10 nm or less in diameter. Considered in connection with the other result, the breakdown of cement paste by

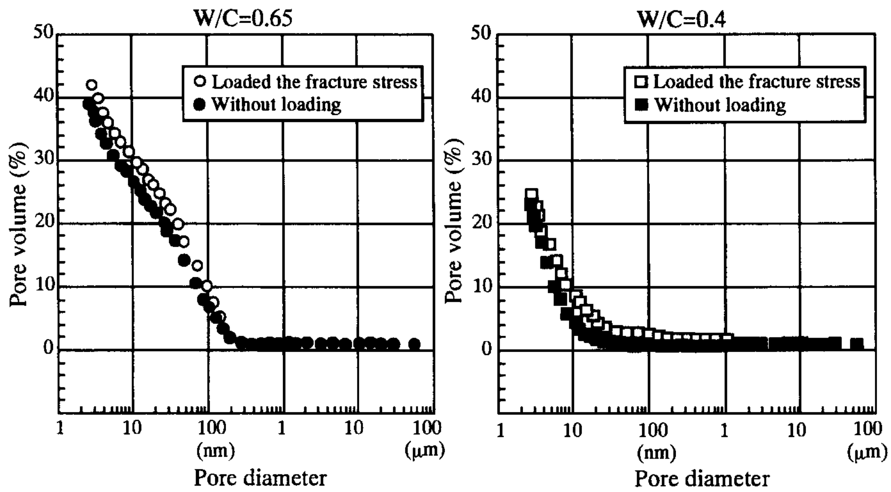



**FIGURE 4.** Cumulative pore volume of normal portland cement paste before and after loading the fracture stress.

the loading of stress occurs in finer texture than in mortar.

Although the volume of pores 30 nm to 1 μm in diameter in blastfurnace slag cement paste prepared at W/C of 0.65 was increased by loading the fracture stress, the difference of the volume of the pores 30 nm or less in diameter before and after the loading of the fracture stress was reduced, and the volume of pores 5 nm or less in diameter was decreased by it. Possibly this is because the pores 30 nm or less in diameter are broken into those 30 nm to 1 μm in diameter. The reason the difference of the pore size distributions in the paste before and after the loading of the fracture stress varies according to the types of cement considered is because the pore structure of hardened paste as well as the magnitude of the fracture stress varies according to the types of cement. It is considered also that the difference in the physical properties derived from the difference in the composition and structure of hydrate composing the paste participates in it. Although the volume of pores 6 nm or more in diameter in the cement paste prepared at W/C of 0.4 was increased by loading the fracture stress and the difference in the volume of the pores 6 nm or less in diameter before and after the loading of the fracture stress was reduced, the difference was smaller than in the paste prepared at W/C of 0.65. The pore diameters of blastfurnace slag cement paste, normal portland cement paste, and fly ash cement paste showing the maximum difference in the pore volume before and after the loading of the fracture stress were 6, 10, and 20 nm, respectively. This trend is the same as those of the magnitude of the fracture stress and its order. This suggests that even finer pores in the cement paste, namely, even the fine region in the texture of it, is broken because of the increase in the fracture stress.

The volume of the pores 60 μm or less in diameter in fly ash cement paste prepared at W/C of 0.65 was increased by the loading of stress, especially 100 nm or less in diameter. This suggests that the generation of fine cracks 100 nm or less simultaneously proceeds with their development into large cracks. Although the volume of pores 500 nm or more in diameter in cement paste prepared at W/C of 0.4 was changed a little by loading the stress, the volume of pores 20 to 500 nm was increased, and the difference in the volume of pores 6 to 20 nm in diameter before and after the loading of stress was reduced although the difference in the volume of the pores 6 nm or less in diameter was increased. The behavior of the cement paste by the loading of stress was similar to that of mortar.

**HARDENED MORTAR AND CEMENT PASTE REPEATEDLY STRESSED.** The changes of cumulative pore volume of normal portland cement mortar and cement paste prepared at W/C of 0.65 caused by the repeated loading of stress are illustrated in Figure 6.

The volume of capillary pores 500 nm or more in diameter in the mortar was increased much more while that of pores 100 to 500 nm in diameter were decreased more by repeated loading of stress at 60% of the fracture stress than by the single loading of the same stress for 1 minute. The volume of capillary pores 100 nm or less in diameter was hardly changed by loading the 60% stress. This suggests that pores 100 to 500 nm in diameter are broken into the pores larger than 500 nm in diameter. The pore size distribution in the cement paste was changed by loading the stress at 60% of the fracture stress in the same manner as by the single loading of the same stress for 1 minute. This phenomenon corresponds to the measurements of the velocity of propagation of supersonic wave.

The changes of the pore structure of hardened mortar

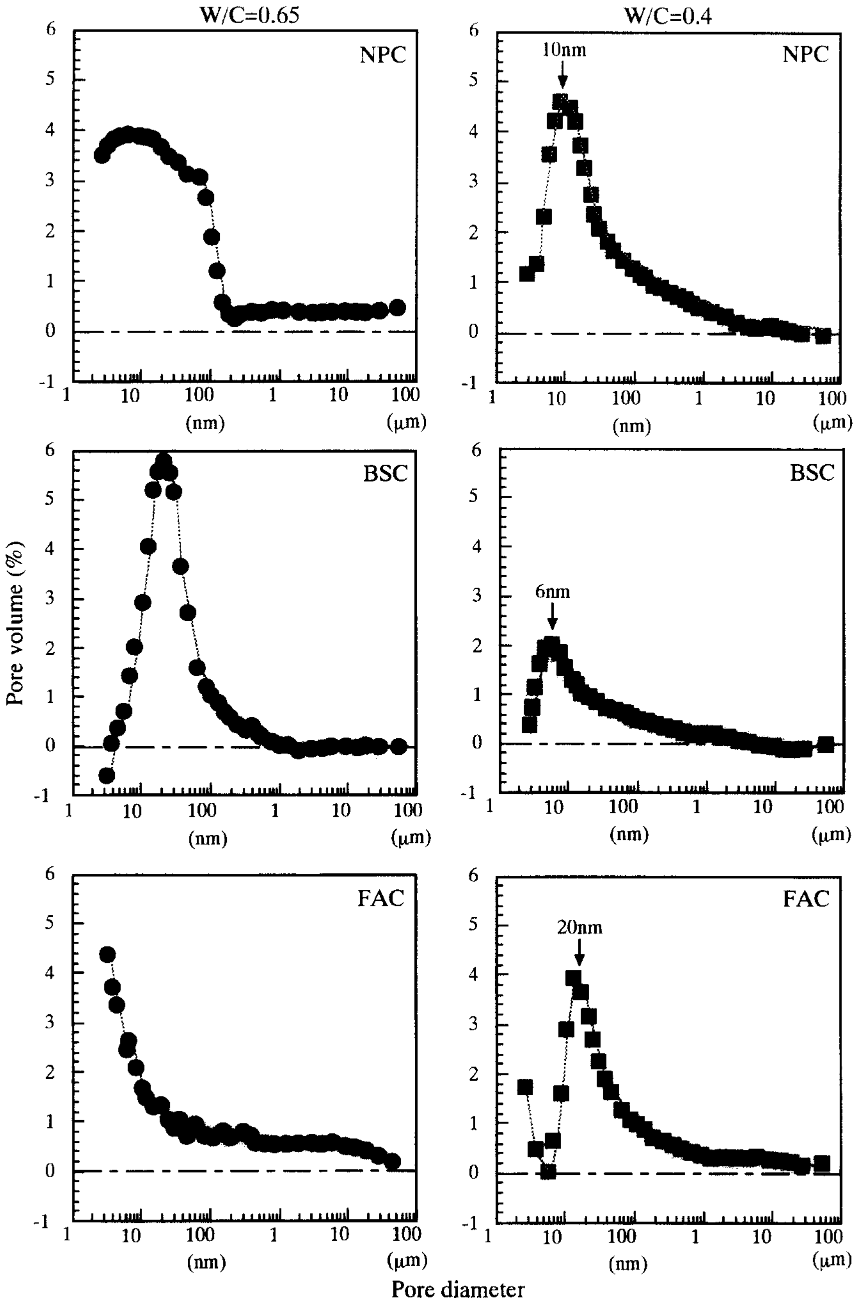



FIGURE 5. Difference of cumulative pore volume of hardened cement paste before and after loading the fracture stress.

and paste prepared from various types of cement by loading the compressive stress are summarized in Table 2.

## Texture and Microstructure

HARDENED MORTAR AND CEMENT PASTE SUBJECTED TO SINGLE STRESS. Figure 7 illustrates the backscattered electron images of the microstructures of hardened mortar and cement paste prepared from various types of cement after loading of the fracture stress.

Broad cracks developed through the transition zone around the aggregate in normal portland cement mortar prepared at W/C of 0.65. Many cracks developed around the aggregate at relatively narrow intervals parallel to each other in normal portland cement mortar prepared at W/C of 0.4. Cracks developed along the interface between aggregate and cement paste, and the fine cracks were observed in the cement paste part of this cement mortar. Most of cracks were prevented from connecting to each other by the aggregate. It is considered that the cracks that developed in the transition zone and the surface of aggregate were enlarged and connected to each other so as to surround the aggregate upon the increase of loading stress, and the test specimen finally collapsed.

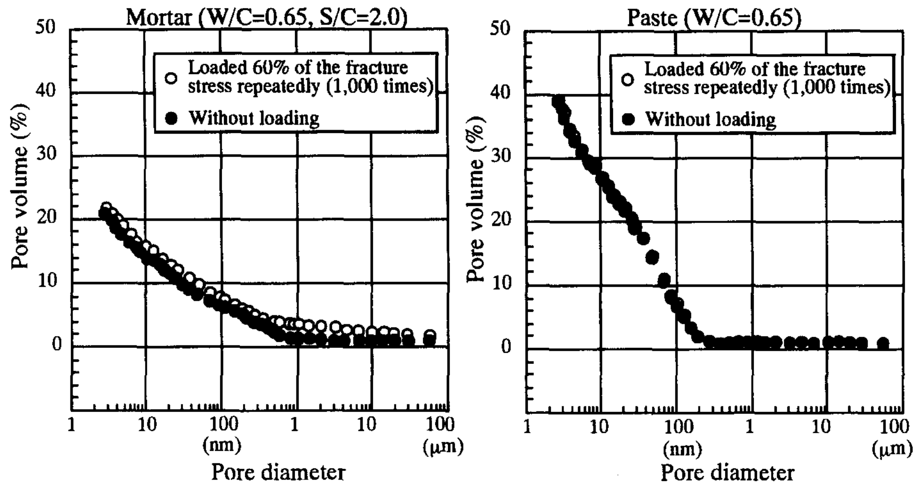



**FIGURE 6.** Cumulative pore volume of hardened mortar and cement paste before and after loading 60% of the fracture stress repeatedly.

It was observed that cracks that extended linearly connected to each other because they were not prevented from propagating by the aggregate in normal portland cement paste. Especially, the fine cracks that developed in normal portland cement paste prepared at W/C of 0.65 extended and widened out, connecting to each other, whereas fine cracks developed in random directions in the peripheral sections of large cracks in that prepared at W/C of 0.4. Few fine cracks were observed in the paste part containing no wide cracks.

Relatively narrow cracks developed through the paste portion apart from the aggregate surface in blastfurnace slag cement mortar prepared at W/C of 0.65 unlike normal portland cement mortar. Fine cracks were observed around the aggregate, while there were relatively few, widely developed cracks in it. The development of cracks was similar to that in normal portland cement mortar prepared at W/C of 0.4. The width of cracks in blastfurnace slag cement mortar prepared at W/C of 0.4 was generally narrower than that in the cement mortar prepared at W/C of 0.65. No wide crack was observed, although a lot of narrow cracks developed in blastfurnace slag cement paste

**TABLE 2.** Change of pore size distribution of hardened mortar (top) and paste (bottom) after loading the fracture stress

| | | | Pore Diameter | | |
|---|---|---|---|---|---|
| | | W/C | Volume Was Decreased | Volume Was Increased | Total Pore Volume |
| Single Loading | NPC | 0.65 | 50–500 nm | 500 nm < | Increase |
| | | 0.4 | 10–50 nm | 100 nm < | Increase |
| | BSC | 0.65 | 10–50 nm | 50 nm < | Increase |
| | | 0.4 | 10–50 nm | 500 nm < | No remarkable change |
| | FAC | 0.65 | 10–100 nm | < 6 nm, 100 nm < | Increase |
| | | 0.4 | 10–500 nm | < 6 nm, 500 nm < | Increase |
| * | NPC | 0.65 | 100–500 nm | 500 nm < | Increase |

| | | | Pore Diameter | | | Pore Diameter Showing Maximum Difference in the Cumulative Pore Volume |
|---|---|---|---|---|---|---|
| | | W/C | Volume Was Decreased | Volume Was Increased | Total Pore Volume | |
| Single Loading | NPC | 0.65 | — | 50–200 nm | Increase | |
| | | 0.4 | < 10 nm | 10–100 nm | Increase | 10 nm |
| | BSC | 0.65 | < 30 nm | 30 nm–1 μm | No remarkable change | |
| | | 0.4 | < 6 nm | 6 nm < | Increase | 6 nm |
| | FAC | 0.65 | — | < 100 nm | Increase | |
| | | 0.4 | 6–20 nm | < 6 nm, 20–500 nm | Increase | 20 nm |
| * | NPC | 0.65 | — | — | No remarkable change | |

*Note*: *Loaded 60% of the fracture stress repeatedly.

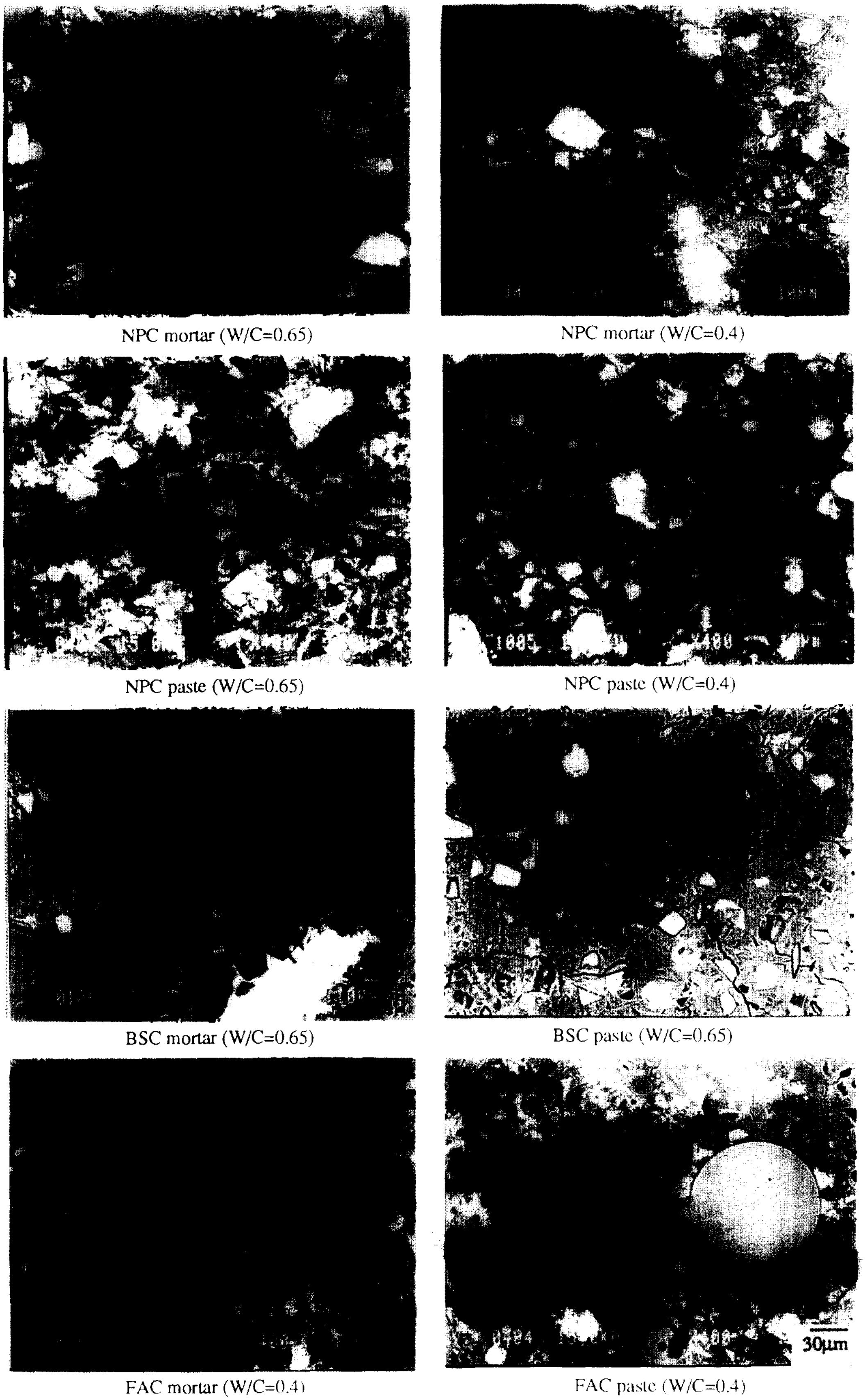
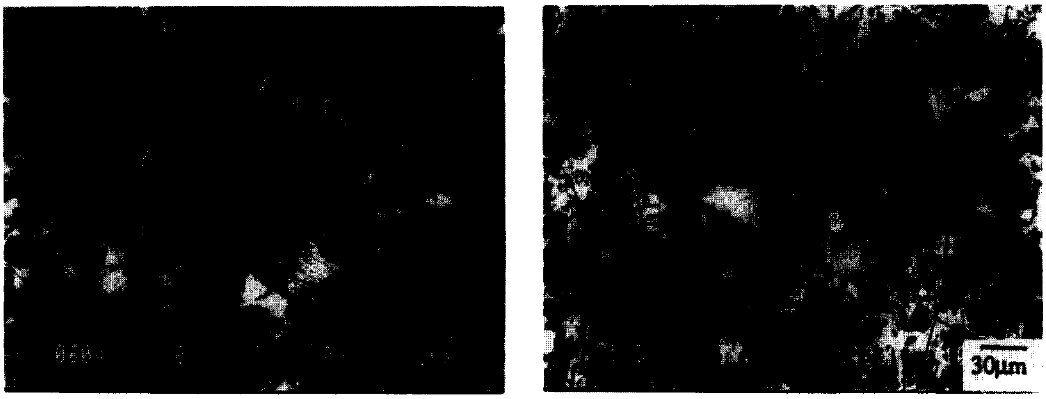



**FIGURE 7.** Backscattered electron images of hardened mortar and cement paste after loading the fracture stress.




NPC mortar (W/C=0.65) NPC paste (W/C=0.65)

**FIGURE 8.** Backscattered electron images of hardened mortar and cement paste after loading 60% of the fracture stress repeatedly (1000 times).

prepared at W/C of 0.65 in the same manner as in the mortar.

In any fly ash cement mortar prepared at W/C of 0.65 and 0.4, $Ca(OH)_2$ and unreacted fly ash remained in the mortar because the pozzolanic reaction hardly proceeded within 28 days and accordingly the texture was porous. Wide cracks developed along the interface between aggregate and cement paste, the surface of fly ash particle, and through the cement paste part. Pores 3 to 6 nm in diameter increased by the loading of the fracture stress determined by the measurement of pore structure could be observed by electron microscopy.

**HARDENED MORTAR AND CEMENT PASTE SUBJECTED TO REPEATED STRESS.** The backscattered electron images of the microstructures of hardened normal portland cement mortar and paste prepared at W/C of 0.65 subjected to 1000 times repeated loading at 60% of the fracture stress are illustrated in Figure 8.

Although many fine cracks developed in normal portland cement mortar, they did not develop into continuous, long, wide cracks. In cement paste, the development of cracks was similar to that in the single loading of stress for 1 minute, and the cracks that developed were narrower than those subjected to the loading of the fracture stress.

The observational results agree with the results obtained by the measurements of the pore size distribution and velocity of propagation of supersonic wave.

## Summary and Conclusions

Using the test specimens of hardened mortar (W/C = 0.65, S/C = 2.0 and W/C = 0.4, S/C = 1.0) and hardened cement paste (mixed with same W/C as mortar) of normal portland cement, blastfurnace slag cement (slag content: 50%), and fly ash cement (fly ash content: 25%) cured at 20°C for 28 days, the changes of texture and pore structure and of the velocity of propagation of supersonic wave by the loading of various modes and magnitudes of compressive stress to those test specimens were examined and the following conclusions were obtained:

1. The compressive strength of the hardened body at the age of 28 days correlated with the porosity of it, and the values of hardened normal portland cement mortar prepared at W/C of 0.65 and 0.4 were 41.3 and 62.7 MPa, respectively, and those of hardened cement paste prepared at the same W/C were 32.6 and 73.6 MPa, respectively. All the values of compressive strength of hardened blastfurnace slag cement mortar and paste, except that of cement paste prepared at W/C of 0.4, were approximately 10% higher than those of hardened normal portland cement mortar and paste. All those values for hardened fly ash cement mortar and paste, except hardened cement paste prepared at W/C of 0.65, were 30% to 40% lower than those of hardened normal portland cement mortar and paste. The effect of the reduction of W/C on the augmentation of strength of hardened fly ash cement mortar and paste was smaller than for normal portland cement and blastfurnace slag cement.
2. The velocities of propagation of supersonic wave in hardened mortar and cement paste subjected to single loading of stress began decreasing slightly by loading the stress higher than approximately 60% of the fracture stress and were sharply decreased by loading the stress higher than approximately 80% of it. The downward trend of velocity

of propagation of supersonic wave accompanied by the increase of the loading of stress was more conspicuous in hardened cement paste than in hardened mortar, in normal portland cement than in blastfurnace slag cement, and in fly ash cement than in normal portland cement. Although the velocity of propagation of supersonic wave in hardened mortar subjected to the repeated loading of stress was lower than that subjected to the single loading of it, both values in hardened cement paste were the same.

3. The pore structures of hardened mortar and cement paste were changed by the loading of stress in such a way that small diameter pores were decreased and large diameter pores and total pore volume were increased, depending upon the pore structure and strength of hardened body before the loading of stress. The destruction of hardened texture by the loading of the fracture stress proceeds, accompanied by breaks of texture around smaller diameter pores in the cement paste than in mortar, in blastfurnace slag cement than in normal portland cement, in normal portland cement than in fly ash cement, and at low W/C than at high W/C.
4. Cracking by the loading of stress starts from structurally weak points and extends in a hardened body. The observational result of backscattered electron images indicated that there were a few fine cracks, although there were many large cracks, in a relatively porous hardened body. This suggests that fine cracks are enlarged rapidly by connecting to each other while breaking the porous texture, such as the transition zone. Meanwhile, many fine cracks were inhibited from developing into large cracks in a hardened body with relatively dense texture. Those results agree with the measurements of the strength, pore size distribution, and velocity of propagation of supersonic wave.

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