



Pergamon

Cement and Concrete Research, Vol. 28, No. 2, pp. 171–176, 1998
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0008-8846/98 \$19.00 + .00

PII S0008-8846(97)00248-2

EXPERIMENTAL STUDY ON THE MICRO-AGGREGATE EFFECT IN HIGH-STRENGTH AND SUPER-HIGH-STRENGTH CEMENTITIOUS COMPOSITES

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(Received September 8, 1997; in final form October 31, 1997)

ABSTRACT

It was experimentally proved in this paper that the micro-aggregate effect played a significant role in both high-strength and super-high-strength cementitious composites with pozzolanic addition by comparing their compressive strength and the parameters of pore structure, and it could make up for the negative influence on strength due to the defect of pore structure. The study demonstrated that the micro-aggregate effect was one of the key factors that contributed to the high strength of DSP materials. © 1998 Elsevier Science Ltd

Introduction

The incorporation of pozzolanic materials is one of the major technical methods for producing high-performance concrete, and the investigation on DSP material in the early 1980s showed that the addition of pozzolanic materials and the compactive cast technique were successful ways to obtain super high-strength cement-based materials (1–3). Therefore, studying the enhancing mechanism of pozzolanic materials is of great importance. In general, the enhancing mechanism is thought to be due to the pozzolanic effect, which involves the micro powder effect, filling effect, pozzolanic reaction effect, and micro-aggregate effect, among which, study on the micro-aggregate effect still remains at a level of understanding its concept.

The micro-aggregate effect may be understood from two aspects, i.e., the effect of particle size and the effect of particle strength of micro-aggregate. The mechanical mechanism of the former effect can be illustrated by the finite element method (4), and the existence of the latter can be testified from the experimental results about the strength and the pore structure in this paper.

Communicated by D.M. Roy.

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TABLE 1
Clinker mineral constituents of Wuyang
brand Portland cement (%).

| C ₃ S | C ₂ S | C ₃ A | C ₄ AF | f-CaO |
|------------------|------------------|------------------|-------------------|-------|
| 55.21 | 21.59 | 4.54 | 16.29 | 0.29 |

Materials and Test Methods

Cement

The cement used in high-strength cementitious materials was a Wuyang brand type Portland cement containing 5% of limestone, its specific surface area being 0.3400 m²/g. The cement used in super-high-strength cementitious materials was a Wuyang brand Portland cement ground to 0.7600 m²/g. The clinker compositions of the two cement are given in Table 1.

Pozzolanic Addition

The chemical constituent and features of silica fume (SF) are shown in Table 2. Both the specific surface area of quartz powder (QP) and quartz glass powder (QGP) are 0.7800 m²/g and the SiO₂ crystal content in quartz powder is 99%. Ultra-fine fly ash (UFFA) has a specific area of 0.7240 m²/g. The chemical constituent and physical properties of UFFA are listed in Table 3 and 4, respectively. It is assumed that the average particle sizes of all the additions are similar on the base of their approximate specific surface area.

Fabrication Method

High-strength cementitious materials were cast with vibration. Super-high-strength cementitious materials were produced at a pressure less than 30 MPa. According to reference (5), so small a pressure had little effect on the initial porosity. However, our observation found that fabricating at such pressure could eliminate visible pores to the naked eye, which was proved to be beneficial to strength development.

TABLE 2
Chemical constituents and characteristic parameters of silica fume*.

| Chemical constituents (%) | | | | | | | BSA***(m ² /g) |
|---------------------------|--------------------------------|--------------------------------|------|------|-----------------|-------|---------------------------|
| SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | MgO | CaO | SO ₃ | LOI** | |
| 91.3 | 1.05 | 0.53 | 1.23 | 0.27 | - | - | 26.0 |

*The SF was provided by Ma'anshan Silica-ferroalloy Plant in China. The average of diameter of the SF was smaller than 0.5 μm.

**LOI is the abbreviation of loss on ignition.

***BSA is the abbreviation of Blaine surface area.

TABLE 3
Chemical constituent of UFFA (%).

| SiO ₂ | Fe ₂ O ₃ | Al ₂ O ₃ | CaO | MgO | SO ₃ | Loss on ignition |
|------------------|--------------------------------|--------------------------------|------|------|-----------------|------------------|
| 51.83 | 5.96 | 32.13 | 3.74 | 1.05 | 0.18 | 1.31 |

Curing Regime

Specimens for autoclave curing were cured in an autoclave for 11 h at 208 and 1.8 MPa pressure after they were kept in room temperature for 24 h after cast. Then, the specimens were kept in room temperature for 1 week before they were finally cured for 24 h in hot air of 200°C.

Mix Proportion

The water-to-solid ratio was 0.24. Solids include cement (C), SF, and UFFA, or QP or QGP. The mix proportion of solids was C:SF:A = 1:0.1:x, where A represents QP or UFFA or QGP, and the value of x is 0.0, 0.25, 0.30, until 0.6. XP-II-type water reducer with a content of 0.5% by weight of solids was applied to make a uniform mixing. The size of specimen was 30 × 30 × 30 mm.

Testing of Parameters of Pore Structure

The testing of distribution was carried out with mercury intrusion porosimetry. The most probable pore radius (a_a) was defined as follows:

$$a_a = \int_0^1 a_i dp_i = \sum_{i=1}^n a_i p_i$$

where a_i is the pore radius of grade i , and p_i is the volume fraction of grade i .

The Micro-Aggregate Effect in High-Strength Cementitious Composites

The Relation Between Particle Size of Pozzolanic Addition and Compressive Strength

Table 5 and 6 listed the compressive strength of specimens that were cured in 1 MPa autoclave and under standard conditions until 28 days, respectively. The content of QP in

TABLE 4
Physical properties of UFFA.

| Specific gravity (g/cm ³) | Ratio of water requirement | Fineness* (%) | BSA (m ² /g) |
|---------------------------------------|----------------------------|---------------|-------------------------|
| 2.42 | 0.88 | 2.36 | 0.7240 |

*Fineness stands for the value of sieve residue of mesh with 45 μm pores.

TABLE 5
Influence of Blaine surface area of QP on
compressive strength of specimen (1 MPa
autoclave, W/S = 0.24).

| BSA (cm ² /g) | 1100 | 3200 | 7800 |
|----------------------------|-------|-------|-------|
| Compressive strength (MPa) | 115.6 | 121.4 | 125.9 |

each specimen was 25%. It can be seen that compressive strength increases with the decline of particle size, which is characterized by specific surface area, under both curing conditions.

Obviously, the increase of the specific surface area of QP is good for playing its role of filling and pozzolanic effect under autoclave. Therefore, the influence of particle size should involve the three different effects.

The Effect of Pozzolanic Addition on Strength

From Table 7 and 8, it is known that the pore distribution of series (C + QP) isn't as good as that of series (C + SF). The proportion of large pores of the former is relatively higher than that of the latter, though their general porosity shows a little difference. From Table 6, however, we can find that the compressive strength of the former (145.1 MPa) is 20 MPa higher than that of the latter. Comparing the results of pore distribution and compressive strength of series (C + SF + QP) with series (C + SF + QGP), the same phenomena can be observed. Although the strength of the addition particles is not available at present, by comparing the strength and the parameters of pore structure, it can be found that cementitious composite with a less-reactive addition such as QP and UFFA possess higher compressive strength than those with a higher-reactive addition such as SF and QGP with same contents under same producing condition, although the pore structure of the former is not necessarily better than the latter. This phenomenon may prove the existence of the micro-aggregate effect of pozzolanic addition.

The Micro-Aggregate Effect in Super-High-Strength Cementitious Materials

The results in Table 9 show that the compressive strength of series (C + SF + QP) is much higher than the other two series. From Table 10, it can be seen that the parameters of pore

TABLE 6
Influence of Blaine surface area of QP on
compressive strength of specimen (standard
curing for 28 days, W/S = 0.24).

| BSA (cm ² /g) | 1100 | 3200 | 7800 |
|----------------------------|------|-------|-------|
| Compressive strength (MPa) | 86.3 | 102.6 | 110.1 |

TABLE 7
Influence of SF, UFFA, QP, QGP, and their composites on
compressive strength (1.5 MPa autoclave, W/S = 0.24).

| Specimen series | Amount of replacement of cement (%) | | | | Compressive strength (MPa) |
|-----------------|--|------|-----|-----|-------------------------------|
| | SF | UFFA | QP | QGP | |
| C | 0 | 0 | 0 | 0 | 102.5 |
| C + SF | 15 | 0 | 0 | 0 | 125.8 |
| C + UFFA | 0 | 15 | 0 | 0 | 119.7 |
| C + QP | 0 | 0 | 15 | 0 | 145.1 |
| C + SF + UFFA | 7.5 | 7.5 | 0 | 0 | 143.3 |
| C + SF + QP | 7.5 | 0 | 7.5 | 0 | 157.6 |
| C + SF + QGP | 7.5 | 0 | 0 | 7.5 | 150.8 |

*The regime of autoclave was 1.5 MPa, 203 and 8 h.

structure of series (C + SF + QP) are not as good as that of series (C + SF + QGP), however, the compressive strength of the former (297.3 MPa) is 50 MPa higher than that of the latter (245.3 MPa). Compared with the case of high-strength cementitious materials, the micro-aggregate effect in super-high-strength cementitious materials is even evident.

Conclusions

The micro-aggregate effect plays its role in both high-strength and super-high-strength cementitious materials and its existence makes up for the negative effect on strength due to the defect of pore structure. Therefore, the role of the micro-aggregate effect is of great importance for DSP materials to have high strength.

TABLE 8
Pore structure parameters of high strength cementitious materials (1.5 MPa and 203
autoclave for 8 h, W/S = 0.24).

| Specimen | Porosity (%) | Distribution of pore radius (%) | | | Most probable pore radius (nm) |
|-------------------------|-----------------|---------------------------------|------------|-----------|--------------------------------------|
| | | <10 nm | 10 ~ 25 nm | >25 nm | |
| C | 16.80 | 54.41 | 41.50 | 4.09 | 22.675 |
| C + 0.15 SF | 14.18 | 57.61 | 40.47 | 1.92 | 13.690 |
| C + 0.15 UFFA | 17.89 | 53.24 | 41.58 | 5.18 | 17.239 |
| C + 0.15 QP | 13.59 | 52.75 | 43.55 | 3.70 | 17.349 |
| C + 0.75 SF + 0.75 UFFA | 15.63 | 58.22 | 38.63 | 3.15 | 15.244 |
| C + 0.75 SF + 0.75 QP | 14.26 | 54.09 | 41.28 | 4.63 | 14.870 |
| C + 0.75 SF + 0.75 QGP | 12.65 | 58.19 | 38.88 | 2.93 | 13.830 |

TABLE 9
Influence of composites of SF, UFFA, QP, and QGP on compressive strength
(1.8 MPa and 203 autoclave for 11 h, then cured in hot and dry air of
200 for 24 h, W/S = 0.24).

| x | Compressive strength (MPa) | | |
|------|----------------------------|--------------------|---------------------|
| | C:SF:UFFA = 1:0.15:x | C:SF:QP = 1:0.15:x | C:SF:QGP = 1:0.15:x |
| 0.0 | 165.3 | 165.3 | 165.3 |
| 0.25 | 178.8 | 245.8 | 205.8 |
| 0.30 | 190.5 | 246.9 | 216.9 |
| 0.35 | 195.1 | 256.3 | 226.3 |
| 0.40 | 213.1 | 283.1 | 253.1 |
| 0.45 | 217.3 | 297.3 | 245.3 |
| 0.50 | 220.1 | 290.1 | 230.1 |
| 0.55 | 206.3 | 256.3 | 206.3 |

TABLE 10
Pore structure parameters of super-high-strength cementitious materials (cured in
autoclave of 1.8 MPa and 203 for 11 h, then cured in hot and dry air of 200 for 24 h,
W/S = 0.24).

| Specimen | Porosity (%) | Distribution of pore radius (%) | | | Most probable pore radius (nm) |
|-------------------------|--------------|---------------------------------|------------|--------|--------------------------------|
| | | <10 nm | 10 ~ 25 nm | >25 nm | |
| C | 15.95 | 63.87 | 26.03 | 10.10 | 21.161 |
| C + 0.15 SF + 0.45 UFFA | 12.46 | 64.94 | 27.7 | 7.35 | 16.699 |
| C + 0.15 SF + 0.45 QP | 12.30 | 74.86 | 18.45 | 7.69 | 13.426 |
| C + 0.15 SF + 0.45 QGP | 12.15 | 77.34 | 17.45 | 5.21 | 12.031 |

Acknowledgment

The authors would like to thank the National Committee of Natural Science Foundation of China for their financial support. Special thanks are also due to Yan An, graduate at Southeast University, and Qi Chengqing, Doctoral candidate at Southeast University, for their help in this research.

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