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Resistance to Internal Damage and Scaling of Concrete Air Entrained By Microspheres

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Abstract. This paper report the test results of high strength concrete produced with slag cement and air entrained with polymer microspheres in three diameters. The study focused on determining the effects of the microsphere size and quantity on the air void structure and resistance to internal cracking and scaling of the concrete. The resistance to internal cracking was determined in compliance with the requirements of the modified ASTM C666 A method on beam specimens. The scaling resistance in a 3% NaCl solution was determined using the slab test in accordance with PKN-CEN/TS 12390-9:2007. The air void structure parameters were determined to PN-EN 480–11:1998. The study results indicate that the use of microspheres is an effective air entrainment method providing very good air void structure parameters. The results show high freeze-thaw durability of polymer microsphere-based concrete in exposure class XF3. The scaling resistance test confirms that it is substantially more difficult to protect concrete against scaling in the presence of the 3% NaCl solution (exposure class XF4). Concrete scaling is a complex phenomenon controlled by a number of independent factors.

1. Introduction
Two forms of concrete damage need to be considered to provide pavement and bridge concrete with the protection against the attack of frost: its resistance to internal cracking and its resistance to scaling. In theory, good freeze-thaw durability can be attained either by producing very tight concrete with W/C<0.38 or by air entrainment.

Scaling is the most frequent damage observed in concrete pavement and bridge structures exposed to cyclic freezing and thawing. It is manifested by gradual flaking and peeling of the paste or mortar over the depth of several millimetres, often accompanied by the exposure of coarse aggregate grains. The scaling occurs at the high degree of saturation in the surface zone of concrete, especially when the surface is covered with a thin film of water or de-icing salt solution. It is more difficult to protect the concrete from scaling than from internal cracking. Scaling of the concrete surface is a complex phenomenon controlled by a number of independent factors [1].

The primary factor that contributes to variation in concrete surface zone properties if the so-called wall effect [2, 3]. Higher porosity and absorption of the surface zone results from the lower volumetric concentration of coarse aggregate and higher volume of porous paste and mortar. Thus, in the mixture with well-chosen proportions, the binder is present in excessive amount relative to the mortar content. The concrete surface zone quality is affected by:

[End of text]
- sedimentation and segregation phenomena resulting from G-forces and compaction method, can increase the local W/C ratio,

- a set of curing-related factors: temperature, exposure to moisture, water migration out of and into the concrete, affecting the porosity of the paste and formation of micro cracks as a result of shrinkage (capillary shrinkage, drying shrinkage or as a result of the difference between the concrete temperature and ambient temperature), or other service-related factors [3, 4],

- other factors that influence the quality of the aggregate-paste interfacial zone [5]; the quality of this zone is one of the primary factors that decide about the rate and extent of paste scaling from the coarse aggregate grains.

Experience has shown that air entrainment is an essential condition to ensure scaling resistant concrete in the presence of de-icing salts. However, the use of several mineral additives and admixtures in the same mixture may lead to difficulties in maintaining a stable air void structure. Fagerlund [6] described three mechanisms of air void structure instability: air bubbles escape from the concrete, small bubbles disappear (dissolve), bubble coalesce into larger pores. The tests for concrete scaling performed according to Borås method by Petersson [7] show that concretes containing an air entraining admixture are more resistant to damage than those produced in combination with superplasticizer. Concrete mixtures of high workability (containing plasticizers) are sensitive to overvibration, which may be accompanied by segregation of mortar or bleeding on the upper surface of members, or increased content of aggregate in the lower layer of the concrete.

High strength of concrete is dependent on low W/B ratio and thereby high binder content. Thus, due to heat of hydration and shrinkage, mineral additives are used, including ground blast furnace slag, for better tightness and resistance to chemical corrosion. It is difficult to attain adequate freeze-thaw resistance, including the protection from scaling, in concretes with slag, where carbonation proceeds more rapidly. The ground granulated blast furnace slag, just like fly ash, requires a careful selection of air entrainer. The concretes rich in slag are extremely susceptible to damage under joint conditions of frost and presence of de-icing salts [8]. According to Giergiczny, Glinicki et al. [9], the use of cement with increased content of the slag may decrease the total amount of air in the hardened concrete, by more than 3% and increase the spacing factor $T$ by more than 0.1mm. Also, the presence of the slag affects the distribution of air particles through decreasing micropore content by more than 1.6%.

The air void instability-related problems can be eliminated by using dimensionally stable particles of chosen diameters, the so-called microspheres. The microspheres provide uniformly dispersed in concrete air voids with adequate and time-independent dimensions. This innovative solution eliminates the basic challenges associated with the coalescence and size variability of air bubbles. The purpose of the study was to determine the effect of different diameter microspheres on the air void structure and freeze-thaw resistance (internal cracking and scaling) of concrete.

2. Materials and Methods

Six series of high strength polymer microsphere air-entrained concrete were manufactured for testing. The microspheres had the diameters of 20, 40 and 80 µm. The tests aimed at determining the influence of the size and quantity of the microspheres on the formation of air void structure and internal resistance of concrete to freezing and thawing and to scaling. The polymer microsphere-based admixture was added at two levels.

The concrete was made up of the following constituents:

- cement CEM II/A-S 42,5N (C),
- silica fume (SF),
- natural sand 0 \(\div\) 2 mm (S),
- coarse aggregate - basalt fraction 4 \(\div\) 8, 8 \(\div\) 16 mm (50:50\%) (B),
- polycarboxylate superplasticizer,
- polymer microspheres (MS-P), D = 20 µm, D = 40 µm, D = 80 µm,
- anti-foaming agent.

The concrete mixture tests were performed for consistency (slump test) (SL) and density (\(\rho_b\)). The hardened concrete tests were carried out for compressive strength (\(f'_{cm}\)), water absorption (\(n_w\)) and freeze-thaw durability – the resistance to internal cracking in accordance with the modified ASTM C666 A test method on beam specimens and the resistance to scaling in the presence of a 3\% NaCl solution to PKN-CEN/TS 12390-9:2007 [10], and porosity characteristics (\(A, A_{300}, \bar{L}\)) in compliance with the requirements set out in PN-EN 480–11:1998 [11].

The specimens were cured in water for seven days and then allowed to air-dry for 21 days. The strength and absorption were tested on 10x10x10 cm concrete cubes. The compressive strength of concrete was determined to PN-EN 12390-3:2011 [12] and water absorption to the Polish standard, PN-88/B-06250 [13].

The resistance of concrete specimens to freezing and thawing was determined in accordance with the modified ASTM C666 A method on 80x80x340 mm concrete beams. After a 28-day curing period, the specimens were immersed in water for seven days and then stored in water-filled metal containers. The specimens were subjected to 300 freeze-thaw cycles.

The scaling resistance of the concrete surface was determined on slabs (the modified slab test) according to PKN-CEN/TS 12390-9:2007 [10]. The test uses a qualitative evaluation of the mass of scaling from the upper surface of the specimen after 7, 14, 28, 42 and 56 cycles of freezing and thawing at the presence of 3\% NaCl solution. Four 50x150x150 mm slabs were tested in each of the six series. The tests were conducted on the natural finished surface of the concrete, perpendicular to the moulding direction. Then, the specimens were stored, prepared and tested following the standard methods. The mass of the scaled material after 56 cycles, in kg/m\(^2\), relative to the surface of the specimen is taken as the test result.

The preparation of polished sections and the determination of porosity characteristics by traverse analysis were performed to PN-EN 480-11:1998. Automatic image analysis was made with the use of a setup consisting of a stereoscopic microscope, a CCD camera and a scanning table.

Table 1 summarises the compositions and selected properties of the concretes.

| Series | Microsphere size | C kg/m\(^3\) | SF kg/m\(^3\) | W/B | S kg/m\(^3\) | B kg/m\(^3\) | MS-P % m.b. | \(\rho_b\) kg/m\(^3\) | SL cm |
|--------|-----------------|-------------|--------------|-----|-------------|-------------|-------------|----------------|------|
| G1     | MS-P20          | 370         | 30           | 0.41| 634         | 1347        | 0.86        | 2551           | 22.0 |
| G2     | MS-P20          | 370         | 30           | 0.42| 633         | 1346        | 1.11        | 2549           | 25.0 |
| G3     | MS-P40          | 371         | 30           | 0.41| 635         | 1349        | 0.86        | 2553           | 22.0 |
| G4     | MS-P40          | 367         | 29           | 0.42| 628         | 1334        | 1.11        | 2526           | 23.0 |
| G5     | MS-P80          | 373         | 30           | 0.41| 639         | 1358        | 0.86        | 2572           | 21.0 |
| G6     | MS-P80          | 369         | 29           | 0.42| 631         | 1341        | 1.11        | 2540           | 22.0 |

Denotation: MS-P – polymer microspheres 20, 40 or 80 µm.
3. Results and discussions

The results of the hardened concrete tests are compiled in Tables 2 and 3. The compressive strength at the age of 28 days ranges from 74.4 to 80.8 MPa. The absorption results are within the 2.40-2.55% range.

Table 3 summarises the results of the air void structure tests. The application of the microspheres reduced the spacing factor $\overline{L}$ to less than 0.20 mm. The $\overline{L}$ values ranged from 0.10 to 0.15 mm. The content of micropores $A_{300}$ was from 1.28 to 2.83%. With fine air bubbles, it is not necessary to obtain the micropore quantity $A_{300} > 1.5\%$. The air content $A$ ranged from 2.01 to 4.35%. The presence of anti-foaming agent in the polymer microsphere-based concretes eliminates the macropores (>300 μm) and provides the total air content of less than 4%.

The changes in mass and length of the beam specimens after 300 freeze-thaw cycles in water are shown in Figure 1 and in Figure 2. No significant changes were observed. All the series tested can be thus regarded as fully resistant to internal damage.

Figure 3 shows the changes in specimen mass after 56 cycles in the 3% NaCl solution. Series G1 and G2 specimens made with polymer microspheres $D=20 \mu m$ showed good ($d_{m56}=0.454 < 0.5 \text{ kg/m}^2$) and sufficient ($d_{m56}=1.333 < 1.0 \text{ kg/m}^2$) resistance to scaling. The specimens made with larger microspheres ($D=40 \mu m$, $D=80 \mu m$) had insufficient scaling resistance in the 3% NaCl solution (mass loss in the range 1.204-1.436 kg/m²). The microspheres with the smallest diameters ($D=20 \mu m$) guarantee that the tiniest and best distributed pores will be provided at a similar total air content. It has to be noted, however, that the test used was the modified slab test in which the specimens were finished, not sawn. Thus, natural surfaces, corresponding to those of the real structure, were tested. Due to different properties of the surface zone, other factors, in addition to air entrainment, affect scaling resistance of concrete. The main reason for diversity of concrete properties in the near surface zone is the wall effect. Less damage (scaled mass) is observed on sawn surfaces than on natural finished surfaces.

### Table 2. Summary of the test results for hardened concretes

| Series | $n_w$ | $f'_{cm28}$ | $f'_{cm56}$ | $d_m$ | $d_L$ | $d_{m56}$ |
|--------|-------|-------------|-------------|-------|-------|-----------|
| G1     | 2.40  | 75.8        | 82.2        | 12    | -0.025| 0.454     |
| G2     | 2.55  | 75.5        | 78.2        | 10    | 0.045 | 0.550     |
| G3     | 2.54  | 80.8        | 82.7        | 7     | 0.01  | 1.436     |
| G4     | 2.43  | 74.3        | 81.0        | 5     | -0.005| 1.333     |
| G5     | 2.43  | 77.3        | 83.2        | 6     | 0     | 1.204     |
| G6     | 2.45  | 75.0        | 82.0        | 9     | -0.01 | 1.353     |

### Table 3. Results of the air void tests

| Series | $A$ | $A_{300}$ | $A_{300}/A$ | $\alpha$ | $\overline{L}$ |
|--------|-----|-----------|--------------|-----------|----------------|
| G1     | 2.01| 1.28      | 64           | 57.44     | 0.13           |
| G2     | 3.67| 1.60      | 44           | 39.24     | 0.15           |
| G3     | 3.06| 1.68      | 55           | 54.86     | 0.12           |
| G4     | 4.35| 2.83      | 65           | 55.44     | 0.10           |
| G5     | 3.64| 2.60      | 71           | 55.17     | 0.11           |
| G6     | 3.89| 2.61      | 67           | 48.27     | 0.12           |
Figure 1. Mass change in beam specimens after 300 cycles of freezing and thawing

Figure 2. Change in length of beam specimens after 300 cycles of freezing and thawing
4. Conclusions

On the basis of the results obtained from the tests of six series of concrete made with slag cement and air entrained with polymer microspheres of three different diameters, the following conclusions were formulated:

1. The use of polymer microspheres is an effective air entrainment method capable of providing very good air void structure parameters ($L = 0.10 - 0.15$ mm).

2. The results of internal deterioration tests performed on beam specimens subjected to freezing in water indicate very good freeze-thaw resistance of concrete air entrained with the addition of polymer microspheres (exposure class XF3).

3. Scaling resistance test results confirm that securing the concrete against surface scaling is much more difficult in the presence of a 3% NaCl solution (exposure class XF4). Scaling of concrete is a complex phenomenon controlled by a number of independent factors. The concrete air entrained by polymer microspheres with the smallest diameters ($D=20\mu$m) satisfied the scaling resistance requirement ($m_{56}<1.0$ kg/m$^2$). The microspheres with the smallest diameters ($D=20\mu$m) provide the tiniest and best distributed voids. It has to be noted, however, that the test used was the modified slab test. Finished test surfaces were used instead of sawn surfaces. Due to different properties of the surface zone, other factors, in addition to air entrainment, affect scaling resistance of concrete. Less damage (scaled mass) is typically observed on sawn surfaces than on natural finished surfaces.

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