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Cement and Concrete Research 35 (2005) 1014–1016

Cement and Concrete Research

Communication

The patch microstructure in concrete: effect of mixing time

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Received 11 May 2004; accepted 15 October 2004

Abstract

It has been previously shown by backscatter-mode scanning electron microscopy (SEM) that various laboratory- and field-mixed concretes exhibit dense areas or patches of hardened cement paste (hcp) alternating with highly porous areas or patches. The present work represents an effort to establish whether this distinctive microstructure was a result of inadequate mixing. A conventional laboratory concrete was prepared and subjected to prolonged mixing in an efficient pan mixer, with small samples being removed periodically, compacted, and cured for 28 days. Examination indicated that evidences of the patchy microstructure persisted despite prolonged mixing for up to 30 min, far beyond normal concrete mixing times.

Keywords: Microstructure; Cement paste; SEM; Backscattered electron imaging; Fresh concrete

1. Introduction

Evidence was recently presented [1] that some field- and laboratory-mixed concretes of water/cement (w/c) ratios of 0.45 or more exhibited a patchy hardened cement paste (hcp) microstructure. Backscatter scanning electron microscopy (SEM) examination revealed distinct areas (patches) of highly porous hcp intermingled with dense areas displaying little detectable porosity. The dense areas often showed considerable populations of residual unhydrated cement; the porous areas comparatively few. Sharp boundaries between the two types of area were frequently observed. Similar alternating patches of dense paste and porous paste had previously been detected in laboratory mortars [2] that had been cited some years ago as evidence for the now-conventional model of the interfacial transition zone (ITZ) [3].

Among the obvious questions raised by these observations was whether or not these features were the result of the inadequate mixing. The laboratory mortars referred to earlier [2] had been unconventionally mixed in a reciprocating shaking device. The field concretes examined had been conventionally mixed in a central ready-mix plant and delivered to the job site in the usual concrete mixer trucks. The present communication reports the results of a straightforward investigation to determine whether prolonged mixing would eliminate the dense area—porous area patchy structure.

2. Materials and methods

A conventional 0.50 w/c concrete was produced for this investigation, using an ASTM Type I cement, crushed dolomite coarse aggregate, and a local river sand of heterogeneous character. No admixtures were used. The batch volume (39 L) was sized appropriately for the open pan countercurrent Lancaster Type SKC mixer used (optimum rated capacity 42 L). The mixing sequence was conventional: coarse aggregate, sand, and cement were added, respectively, and dry mixed for 2 min. Water was then added while the mixer continued rotating, the stated mixing time starting from the completion of the water addition.

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doi:10.1016/j.cemconres.2004.10.023

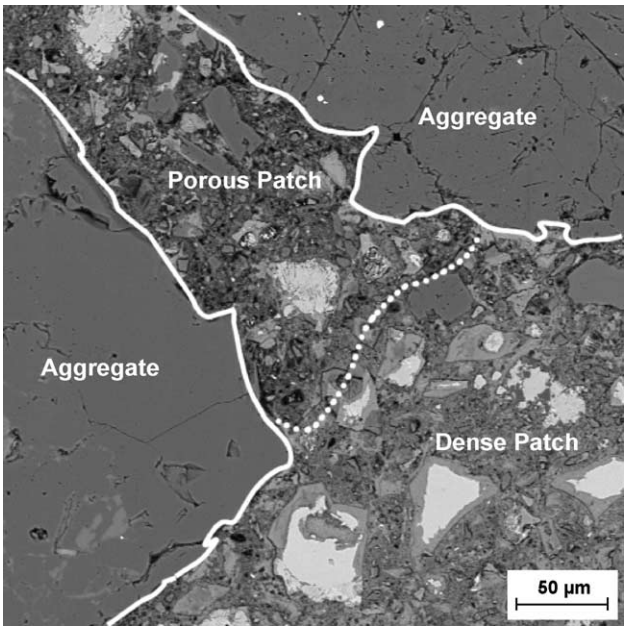

Fig. 1. Adjacent porous and dense patches in concrete after 1 min mixing. Note the content of bright residual cement grains in the dense patch.

Mixing was continuous except for brief interruptions to permit small samples of the fresh concrete to be collected at 1, 2, 3, 5, 10, 15, 30, and 45 min. Each sample was consolidated on a vibrating table, sealed for 24 h, and then exposed to a fog room for 27 additional days. Laboratory temperature was approximately 23 °C throughout.

After the 28 days of curing, thin prisms were sawn from the center portions of each specimen using a precision diamond saw and a nonaqueous lubricant. They were then dried, impregnated with epoxy resin, hardened, and finally polished in the usual manner for backscatter SEM examination.

The slump and the temperature of the fresh concrete were measured during the brief (1 to 2 min) interruptions of mixing needed to obtain the samples.

3. Results

3.1. Effects of prolonged mixing on the characteristics of the fresh concrete

The slump after 1 min of mixing was 160 mm. It increased slightly then declined on further mixing to 70 mm at 15 min, 20 mm at 30 min, and to 0 mm at 45 min. At 45 min, the mix had developed sufficient stiffness to stand upright and maintain an edge produced by a cutting tool. Consolidation of the 30-min and especially the 45-min samples was difficult. However, the temperature of the fresh concrete mass was hardly affected by the prolonged mixing, being between 8 and 9 °C throughout.

3.2. Microstructural effects

Examination of the 1-min specimen revealed the patchy microstructure previously described [1]. Fig. 1 shows an area displaying neighboring porous and dense patches, with the boundary between them particularly sharp. The dense area shows a substantially higher content of residual cement grains than the porous area, with inner product C–S–H surrounding most of them.

The division of the hcp into dense and porous areas persists in Fig. 2, taken from the 10-min specimen. The darker appearance of the porous patch (due to epoxy-filled pore space) is evident, and unhydrated cement grains are virtually absent within it. Figs. 2 and 3 are shown in the two-image format provided by the Aspex Personal SEM

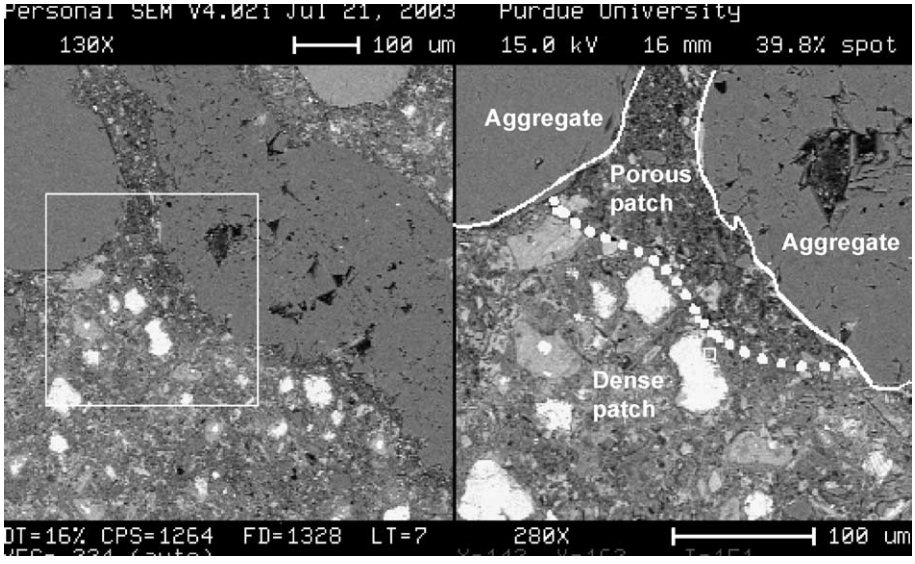

Fig. 2. Area showing the persistence of adjacent dense and porous patches after 10 min of mixing.

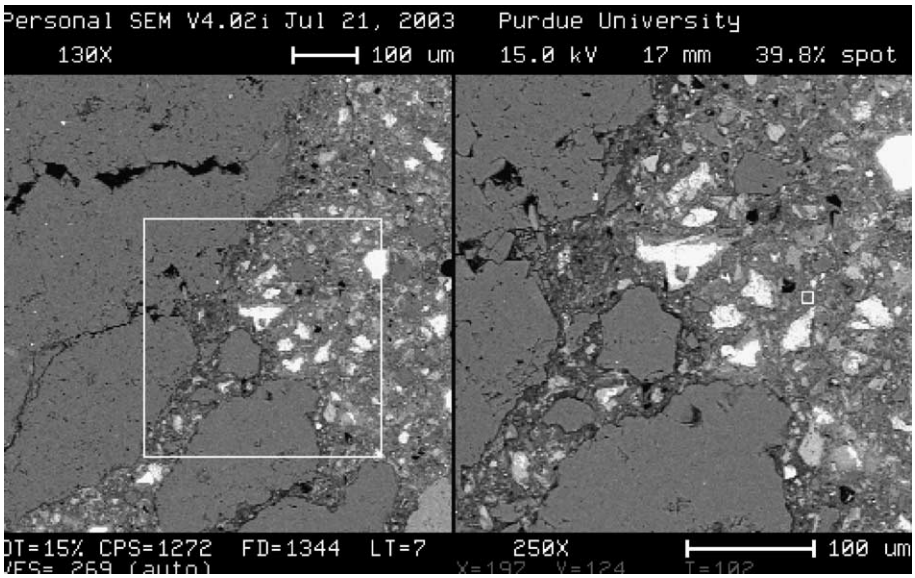

Fig. 3. Area indicating the continued persistence of the patchy structure after 30 min of mixing.

used in this investigation. The upper scale bar pertains to the lower-magnification image on the left. The lower scale bar pertains to the area depicted in higher magnification on the right, which is outlined in the square box.

Fig. 3 is taken from a specimen obtained after 30 min of mixing and confirms that evidences of the patchy structure are still present despite this prolonged mixing.

The microstructure of the concrete specimen obtained after 45 min of mixing was significantly different. The hcp appears dense throughout but contains a substantial proportion of dark 40- to 60-μm grains not seen previously. EDS spectra indicated that these were dolomite. It appears that, in the extremely prolonged mixing, part of the dolomite coarse aggregate was ground to cement fineness and incorporated within the fresh cement paste, modifying its microstructure substantially. The extreme stiffening of the fresh concrete noted at 45 min (zero slump, ability to support vertical surfaces) is apparently a consequence of the production of these dolomite fragments and their incorporation as filler into the fresh paste.

In any event, it is apparent that evidences of the patch microstructure persist for at least 30 min of highly effective mixing. Accordingly, the patchy structure is deemed not to be an artifact of casual or incomplete concrete mixing.

4. Conclusions

(1) The patchy structure of porous hcp areas interspersed with dense bright hcp areas of little visible porosity reported previously in backscatter SEM observations of a laboratory mortar and field concretes is also found in a conventional laboratory mixed concrete of normal w/c (0.5).

(2) This patchy structure appears to be maintained under prolonged mixing for as much as 30 min. Thus, its occurrence does not arise as an artifact of insufficient concrete mixing in the specimens or concretes previously examined.

(3) For extremely prolonged (45 min) mixing in the present concrete, dolomite fragments were produced from the coarse aggregate and incorporated in the hcp, substantially modifying its microstructure and rheological character.

Acknowledgements

I thank Mark Baker for assistance in the concrete batching and mixing, Sulapha Peethamparan for preparation of the SEM specimens and assistance with scanning electron microscopy, and Jan Olek for perceptive discussions.

References

[1] S. Diamond, The patchy structure of cement paste in conventional concretes, ‘Concrete Science and Engineering: A Tribute to Arnon Bentur’, RILEM Proc. PRO 36, RILEM Publications S.A.R.L, Paris, 2004, pp. 85–94.

[2] S. Diamond, Percolation due to overlapping ITZs in laboratory mortars? A microstructural evaluation, Cem. Concr. Res. 33 (7) (2003) 949–955.

[3] D.N. Winslow, M.D. Cohen, D.P. Bentz, K.A. Snyder, E.J. Garboczi, Percolation and pore structure in mortars and concrete, Cem. Concr. Res. 24 (1) (1994) 25–37.